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CANADIAN WILTSHIRE BACON

XXIV. EFFECT OF STRONG CURES ON KEEPING QUALITY¹

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Abstract

The effect on keeping quality of hard curing by both wet and dry salting methods, and of treatment with borax, boric acid, acetylsalicylic acid, and a mixture of benzoic and citric acids was investigated on sides and gammons stored at 15.6° C. (60° F.) for periods up to 40 days. Of the methods studied for preventing spoilage, packing in a 13 : 3 salt-borax mixture was the most effective, but would be detrimental to flavour quality because of the presence of excessive quantities of sodium chloride and undesirable desiccation of the meat.

Experiments on the effect of surface bacterial contamination on internal spoilage indicated that deterioration in the deep meat was relatively independent of the condition of the surface.

Introduction

During the early war period an anticipated shortage of refrigerated space for the shipment of Wiltshire bacon to England prompted investigations on curing, smoking, and chemical preservative treatments that might prevent deterioration over extended periods at ordinary temperatures. The results of certain studies on smoking and chemical preservatives have been reported or are in press (9, 11, 12, 13). The present paper describes four experiments on the suitability of strong cures and other supplementary treatments for maintaining quality at elevated temperatures. In addition, observations on the effect of surface bacterial contamination on internal spoilage of bacon are given.

I. Tank Curing and Surface Treatment with Borax and Boric Acid

PROCEDURE

It was desired to ascertain the relative effect of strong, or so-called 'hard', tank cures, borax, and boric acid, on keeping quality. Since a saturated solution of sodium chloride is normally employed in the curing of Canadian Wiltshire bacon, it was necessary to use other methods to obtain the required

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hard-cured product. Of the available methods extra pumping, overhauling, and increasing the ratio of pickle to meat were considered most suitable for use in this study. Three sides, taken from different hogs, were cured by each of the following procedures: (i) hard—commercial pumping pickle and procedure; cured four days in a small tank; overhauled; repumped; and cured for a further four days; (ii) medium—commercial pumping pickle and procedure, and cured eight days in a small tank; and (iii) regular—as for (ii) but cured eight days in a large commercial tank. The meat to pickle ratio in the small tank was approximately 5 lb. of meat to 1 gal. of pickle as compared to the usual proportion of about 20 lb. to 1 gal.

After removal from cure the sides were drained for one day at 3.3°C . (38°F .) and wiped, prior to baling. The meat surface of three sides, representing each of the types of cures, was dusted with 1 lb. of borax, another set of three each with 1 lb. of boric acid, and the remainder left untreated. Thus all combinations of cure and surface treatment were studied. The three sides given the same surface treatment were baled together so that meat to meat surfaces were together for two of the sides, and meat to skin for the third. The bales were stored at 15.6°C . (60°F .) for 32 days, conditions considered to be comparable to those encountered in shipment as stowage. The entire experiment was duplicated with a second group of nine sides.

In tests of this nature it was considered unnecessary to adjust the quantities of borax and boric acid to a comparable basis in terms of boron, since treatments of 1 lb. per side represented an excess of both compounds. Moreover, losses from the surface due to mechanical action, subsequent drainage of the solution formed with the meat juices, and the differential solubility of the two compounds vitiate comparisons based on boron content.

Bacteriological, chemical, and visual examinations of the sides were made at the end of the storage period. Chloride, nitrate, nitrite (7) and, in certain instances, borax or boric acid determinations (1, p. 460) were made on a slice of gammon approximately 1.25 in. thick, removed from immediately in front of the round bone, and on a sample of the back between the third and sixth ribs.

Differences in the sodium chloride content and, for certain samples, the borax and boric acid content, between the inside position adjacent to the fat and the surface layer were determined on a central strip of a slice of the gammon, approximately 1.5 in. in thickness, containing the round bone (10). Chloride was determined by a wet oxidation method (6). Peroxide oxygen determinations were made on the gammon fat (8).

Bacteriological counts were made of the deep meat of all gammons, and the rib surfaces for most of the sides. The deep-meat samples were taken from a slice of the gammon approximately 2 in. thick that had been flamed on both sides until charred. Several cores approximately 0.5 in. in diameter were obtained with a stainless steel cork borer, transferred to a Petri dish, and the charred and coagulated meat from each core removed. A sample of about 5 gm. was ground with sand, diluted with a 4% sodium chloride solution, and plated on 4% salt agar. Counts were made after incubation for six days at

20° C. Surface counts were made on samples of 9 to 12 sq. cm. of rib surface by a procedure described previously (3).

RESULTS

Visual Examination

Visual examination of the sides after storage for 11 days at 15.6° C. showed them to be in a reasonably satisfactory condition. After 32 days' storage no obvious differences could be detected between the three cures (Table I). Of

TABLE I

EFFECT OF TYPE OF CURE AND SURFACE TREATMENT ON THE VISIBLE APPEARANCE AND CONDITION OF WILTSHIRE SIDES AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure ¹	Surface treatment		
	None	Borax	Boric acid
Hard	Very slimy; gammon and pocket sour; very moist. Poor condition.	Slime; excess salt; moist; pocket sour. Fair condition.	Slight slime on ribs; mould; slight excess salt; moist; fore end slightly sour. Fair condition.
Medium	Very slimy; gammon and pocket sour; very moist; one side with gas on ribs and blue stain. Poor condition.	No visible slime; excess salt; slightly moist; one gammon gassy and sour. Fair condition.	Slight slime on ribs; mould; slight excess of salt; slightly moist. Fair condition.
Regular	Very slimy; gammon sour; very moist; one side with blue stain on ribs; surface sour. Poor condition.	Slime; excess salt; moist; gammon sour. Fair condition.	Slight slime on ribs; moulds; slight excess of salt; slightly moist. Fair condition.

¹ As defined in the text.

the surface treatments boric acid appeared to be more effective than borax. The colour of all the sides was good. All the bales were very wet. The regions of the blade pocket and around the bones appeared to be most vulnerable to spoilage. While the odour of the freshly cut surface of none of the sides was putrid, it was not prime, and suggested the occurrence of incipient changes.

Chloride Content and Distribution

The average chloride content of the hard-cured backs and gammons was greater than that of the medium- or regular-cured product, but the differences were smaller than expected (Table II). A satisfactory distribution of chloride in the sides was obtained for all three sides. Additional pumping effectively introduced sodium chloride into the deeper portions of the gammon.

Nitrate Content

The hard-cured sides contained the most nitrate (Table III). There was little difference between the amount in the back and the gammon. Since a

similar conclusion was noted for the chloride content, it would appear that the pumping and curing procedures employed gave satisfactory distribution of salts in the sides.

TABLE II

EFFECT OF METHOD OF CURE ON THE CONTENT AND DISTRIBUTION OF SODIUM CHLORIDE IN WILTSHIRE BACKS AND GAMMONS AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure ¹	Sodium chloride, %			
	Back	Gammon	Distribution in gammon ¹	
			Inside	Outside
Hard	5.6	6.1	9.0	8.7
Medium	5.5	5.6	7.7	8.1
Regular	5.4	5.3	7.4	7.6

¹ As defined in the text.

TABLE III

EFFECT OF METHOD OF CURE ON THE CONTENT OF SODIUM NITRATE IN WILTSHIRE BACKS AND GAMMONS AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure ¹	Sodium nitrate, %	
	Back	Gammon
Hard	0.27	0.31
Medium	0.19	0.24
Regular	0.22	0.23

¹ As defined in the text.

Nitrite Content

While the mean nitrite content of the gammons over all surface treatments increased with increase in the hardness of the cure, that of the backs decreased (Table IV). The mean nitrite content of both backs and gammons over all cures was greatest for sides treated with borax, less for those given no surface treatment, and least for those on which boric acid had been dusted. This must be attributed primarily to differential growth of nitrate-reducing micro-organisms, since the magnitude of the differences is such that they cannot be accounted for directly by variable pumping procedures. It would appear that the growth of nitrate-reducing micro-organisms was little affected by borax but was retarded by boric acid.

In most instances the mean nitrite content was high. This suggests that the concentrations of nitrate employed in Canadian standard Wiltshire curing pickles should be reduced if bacon is to be held at 15.6° C. for one month without approaching or exceeding the legal limit of nitrite (200 p.p.m.) (2).

TABLE IV

EFFECT OF TYPE OF CURE AND SURFACE TREATMENT ON THE CONTENT OF SODIUM NITRITE IN WILTSHIRE BACKS AND GAMMONS AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure ¹	Surface treatment			
	None	Borax	Boric acid	Mean
	Sodium nitrite, p.p.m.			
<i>Gammon</i>				
Hard	296	664	90	350
Medium	257	281	157	232
Regular	208	404	15	209
Mean	254	450	87	264
<i>Back</i>				
Hard	59	216	83	119
Medium	173	251	89	171
Regular	139	453	7	200
Mean	124	307	60	163

¹ As defined in the text.

Content and Distribution of Borax and Boric Acid

Both the backs and gammons, on the average, contained more boric acid than borax (Table V), a reflection of the relative solubilities of the two compounds. The concentration of borax and boric acid in the gammons was approximately four times greater at the external than at the inside sampling positions. Although the quantities of borax and boric acid observed here are not toxic, it should be recalled that boron acts as a cumulative poison in the human body.

TABLE V

CONTENT AND DISTRIBUTION OF BORAX AND BORIC ACID IN WILTSHIRE BACKS AND GAMMONS AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Back	Gammon	Borax, %		Boric acid, %			
		Distribution in gammon		Back	Gammon	Distribution in gammon	
		Inside	Outside			Inside	Outside
0.45	0.35	0.24	0.97	0.48	0.72	0.35	1.33

Peroxide Oxygen Content

The mean peroxide oxygen content of the gammon fat, averaged over all surface treatments, progressively decreased from the hard-cured to the regular-cured product (Table VI). It would appear that the addition of

TABLE VI

EFFECT OF TYPE OF CURE AND SURFACE TREATMENT ON THE PEROXIDE OXYGEN CONTENT OF WILTSHIRE GAMMON FAT AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure ¹	Surface treatment			
	None	Borax	Boric acid	Mean
	Peroxide oxygen content, ml. 0.002 N Na ₂ S ₂ O ₃			
Hard	7.4	2.6	1.5	3.8
Medium	2.5	2.9	1.4	2.3
Regular	2.0	1.3	1.4	1.6
Mean	4.0	2.3	1.4	2.6

¹ As defined in the text.

further quantities of curing salts was associated with a decrease in the stability of the fat. The mean peroxide oxygen content, as averaged over all types of cures, was greatest for the control, less for borax and least for boric-acid-treated sides. This may be a reflection of the relative effects of the various treatments on the growth of micro-organisms elaborating oxidizing enzymes.

Bacterial Counts

The surface bacteriological counts were high and indicate either the presence or imminent appearance of slime (Table VII). While slime could be readily observed on the untreated surfaces, it was difficult to detect on those dusted with borax or boric acid. The type of cure had little effect on surface bacterial growth. The mean count for the sides treated with borax was slightly higher than that of the controls, showing that borax had little or no bactericidal or bacteriostatic action on surface organisms. Other experiments have indicated as great or greater bacterial development on bacon treated with borax as on controls (4). It is believed that this is due to a raising of the pH of the meat to a level more favourable to bacterial development. The use of boric acid effected some decrease in the mean surface count.

In the deep-meat counts (Table VII) a distinction was made between the large, easily seen type of colony, such as appeared on surface plates, and the small, pin-point types that are just discernible with the naked eye and must be counted at a magnification of 10 or 15 \times . The mean number of large and of pin-point colonies was approximately the same for all types of cure and surface treatments, and showed little relation to the surface count. Pin-point counts were on the average 1000 times higher than those for the large colonies.

TABLE VII

EFFECT OF TYPE OF CURE AND SURFACE TREATMENT ON SURFACE AND DEEP-MEAT BACTERIOLOGICAL GROWTH IN WILTSHIRE BACON AFTER STORAGE FOR 32 DAYS AT 15.6° C.

Type of cure	Surface treatment			
	None	Borax	Boric acid	Mean
<i>Surface</i>				
<i>Log₁₀ number of organisms per sq. cm.</i>				
Hard	8.99	9.05	—	—
Medium	8.17	9.04	6.04	7.75
Regular	8.38	8.80	6.16	7.78
Mean	8.51	8.96	6.10	—
<i>Deep-meat—(a) Large colonies</i>				
<i>Log₁₀ number of organisms per gm.</i>				
Hard	3.43	3.79	3.89	3.70
Medium	3.78	3.00	4.03	3.60
Regular	3.32	3.02	3.39	3.24
Mean	3.51	3.27	3.77	3.51
<i>Deep-meat—(b) Pin-point colonies</i>				
<i>Log₁₀ number of organisms per gm.</i>				
Hard	7.21	6.63	6.74	6.86
Medium	6.12	6.48	7.00	6.53
Regular	7.16	6.95	6.21	6.77
Mean	6.83	6.69	6.65	6.72

II. Combined Tank and Dry Curing; Drainage Time; and Surface Treatment with Benzoic and Acetylsalicylic Acids

PROCEDURE

The experiment was designed to obtain information on the effect of combined tank and dry-salt curing, drainage time, and surface treatment with benzoic acid and acetylsalicylic acids (11) on the keeping quality of bacon. For this purpose the right and left sides of four hogs were pumped and cured, using standard Wiltshire curing pickles, and drained, as shown in Table VIII. The shoulders were removed, and the meat surface of the remaining portion of the sides dusted either with a mixture of 31.5 gm. of benzoic acid and 15.5 gm. of citric acid or with 31 gm. of acetylsalicylic acid. After baling, the sides were stored at 15.6° C. (60° F.), and visually examined after 16 and 32 days.

TABLE VII
EFFECT OF COMBINED TANK AND DRY CURING, DRAINAGE TIME, AND SURFACE TREATMENT WITH BENZOIC AND ACETYLSALICYLIC ACIDS ON CONTENT AND DISTRIBUTION OF CURING SALTS IN, AND BACTERIAL GROWTH ON, WILTSHIRE BACON AFTER STORAGE FOR 32 DAYS AT 15.6°C.

Side No.	Curing treatment	Drainage time	Surface treatment ¹	Gammon				Salt distribution in gammon				Surface bacterial count, log no. organisms per sq. cm.					
				NaCl, %	NaNO ₂ , p.p.m.	NaCl, %	NaNO ₂ , p.p.m.	NaCl, %	NaNO ₂ , p.p.m.	NaCl, %	NaNO ₂ , p.p.m.	I	O	I	O		
1 R	Tank, 8 days	0	Benzoic acid	—	—	—	—	—	—	—	—	—	—	—	3.90	0	
2 R	Tank, 8 days	0	Acetysalicylic acid	—	—	—	—	—	—	—	—	—	—	—	6.63	0	
3 L	Tank, 8 days	1	Benzoic acid	5.72	0.07	18	6.08	0.07	18	6.04	5.73	0.07	0.06	12	33	6.60	0
4 L	Tank, 8 days	1	Acetysalicylic acid	6.20	0.12	28	5.52	0.09	32	5.82	5.66	0.11	0.13	14	33	6.87	0
1 L	Tank, 8 days	3	Benzoic acid	—	—	—	—	—	—	—	—	—	—	—	—	5.31	6.38
2 L	Tank, 8 days	3	Acetysalicylic acid	—	—	—	—	—	—	—	—	—	—	—	—	6.48	0
3 R	Tank, 5 days; dry salt, 4 days	0	Benzoic acid	7.01	0.05	36	6.32	0.09	24	7.12	7.42	0.09	0.09	17	31	5.92	6.72
4 R	Tank, 5 days; dry salt, 4 days	0	Acetysalicylic acid	6.20	0.07	7	7.00	0.15	39	7.22	7.06	0.17	0.13	22	6	9.16	0

I: Position adjacent to fat.

O: Position adjacent to surface.

¹ See text.

At the termination of the storage period, surface bacterial counts were made on all sides, and the content of curing salts in the backs and gammons determined for four of the sides. Measurements were also made of the distribution of curing salts in the gammons. The methods employed have been described in Part I.

RESULTS

No definite changes could be observed after 16 days' storage. However, at 32 days, 3L had a generally sour odour and 1L, 2R, 2L, and 3R were slightly sour in areas adjacent to the round bone (Table VIII). Slime was very heavy on Side 4R, while on the others it was mainly localized around the bones. Moreover, the skin was quite slimy on those sides that were at the bottom of the bales and had remained moist. There was little difference between acetylsalicylic acid and the mixture of benzoic and citric acids as preventatives of slime. However, while moulds were present on all sides, they were especially prevalent on those treated with acetylsalicylic acid.

The results of the chemical analyses on the meat are given in Table VIII. The salt content of the sides given the combined curing treatment was slightly greater than that of sides cured in the tank only, but the differences were smaller than expected and would not merit the extra processing costs involved.

III. Dry Salting of Wiltshire Gammons

PROCEDURE

The investigation was undertaken to assess the suitability of a suggestion often made that Wiltshire bacon could be shipped satisfactorily under stowage conditions if packed with dry salt and borax in wooden boxes. The material studied consisted of four Wiltshire gammons from different hogs, taken over the available size range. To simulate attachment to the original sides, the cut surfaces were dipped in melted vegetable shortening, and further protected by a double thickness of waxed paper. After an initial sampling, two of the gammons were placed in a wooden box and packed in a 13 : 3 salt-borax mixture. The top layer, separated by waxed paper, consisted of the remaining two gammons, which were thus available for interim sampling. One pound of the preservative mixture was used to four pounds of meat. The gammons were stored at 15.6° C. for 40 days, conditions considered to be comparable to a stowage environment.

Differences in the sodium chloride and moisture content between the inside position adjacent to the fat and the outer layer were determined during the storage period at 10-day intervals. For this purpose, two 0.75 in. borings were taken from sites chosen at random on the exposed meat surface, leaving space for the two final cross-sectional slices mentioned below. In order to avoid increased surface exposure of the gammons to the salt mixture, the holes were filled with solid vegetable shortening. Additional preservative was necessary to reproduce initial conditions after each sampling.

The distribution of salt, moisture, and borax at the beginning and end of storage was determined by the central strip method, described in Part I, on slices of the gammon removed from each of two positions, *A*, near the butt end and *B*, near the round bone. Additional slices from each of the two positions were combined in order to obtain some representation of the entire gammon. These complete samples were analysed for their chloride, moisture, and borax content. Spoilage in the gammon fat was assessed by determination of the peroxide oxygen content. Deep-meat bacterial counts were made on the central sections of the two undisturbed gammons.

The methods employed for determination of the deep-meat bacterial counts, and the salt, borax, and peroxide oxygen content have been described in Part I. The moisture content of the meat was determined by drying 2- to 3-gm. samples *in vacuo* for 16 hr. at 100° C.

RESULTS

Organoleptic inspection indicated the freshly-cut meat surfaces to be still in fair condition after 40 days' storage. The surfaces directly exposed to the salt mixture were dark in colour, hard and dry, with no evidence of moulds or slime.

The distribution of salt and moisture after 0, 20, and 30 days' storage is shown in Table IX. As expected, the salt content increased with storage

TABLE IX

AVERAGE CHANGES IN THE DISTRIBUTION OF SALT AND MOISTURE DURING STORAGE AT 15.6° C. OF TWO WILTSHIRE GAMMONS IN A SALT-BORAX MIXTURE

Storage time, days	Sodium chloride, %		Moisture, %	
	Inside	Outside	Inside	Outside
0	2.46	4.68	75.0	67.1
10	4.92	7.62	70.2	65.9
20	7.41	10.43	68.2	59.5
30	8.82	12.16	65.4	54.9

time, while the moisture content decreased. Improvement in distribution with storage was slight. Comparison with Tables X and XI shows that the small borings employed do not represent this type of material with any great efficiency. However, they are of value in indicating trends, as in the present instance.

The results for the distribution of chloride, moisture, and borax at the beginning and end of the storage period are presented in Table X. The salt gradient at the end of storage, while better than it was initially, is not regarded as particularly satisfactory. The salt level differed both across and along

TABLE X

DISTRIBUTION OF SALT, MOISTURE, AND BORAX IN WILTSHIRE GAMMONS AT THE BEGINNING AND END OF STORAGE AT 15.6° C FOR 40 DAYS IN A SALT-BORAX MIXTURE

Storage time, days	Sample No.	Sodium chloride, %				Moisture, %				Borax, %			
		Position A ¹		Position B ²		Position A		Position B		Position A		Position B	
		Inside ³	Outside ³	Inside	Outside	Inside	Outside	Inside	Outside	Inside	Outside	Inside	Outside
0	1	—	—	1.83	8.09	—	—	70.0	65.3	—	—	—	—
	2 and 3	—	—	3.34 ³	5.83	—	—	73.0	68.3	—	—	—	—
	4	—	—	1.45	4.73	—	—	78.4	69.3	—	—	—	—
40	1	9.65	12.39	11.90	13.90	61.6	56.6	60.0	55.4	0.17	0.46	0.25	0.72
	2 and 3	7.30 ⁴	9.52	8.33	10.20	68.6	62.1	68.4	63.9	0.18	0.38	0.19	0.42
	4	7.14	9.81	8.78	10.40	68.2	61.2	65.2	62.2	0.13	0.39	0.16	0.47

¹ Values averaged for Sample Nos. 2 and 3.

² See text.

TABLE XI

CHEMICAL AND BACTERIOLOGICAL MEASUREMENTS ON WILTSHIRE GAMMONS AFTER 40 DAYS' DRY SALTING AT 15.6° C.

Sample No.	Sodium chloride, %	Moisture, %	Borax, %	Peroxide oxygen content of fat, ml. 0.002 N Na ₂ S ₂ O ₃	Deep meat bacterial content, log ₁₀ no. per gm.	
					Large colonies	Pin-point colonies
1	12.71	57.6	0.43	0.94	3.24	6.38
2 and 3	9.02 ¹	65.6	0.29	2.25	—	—
4	9.67	64.1	0.36	1.82	2.38	6.30

¹ Values averaged for sample Nos. 2 and 3.

the gammons, i.e. between the inside and outside layers and Positions A and B. Moisture distribution was as expected, the outer layer being about 5% drier than the inner. When borax was mixed with salt, borax distribution was less than half that when used alone (cf. Table V).

The over-all chloride, moisture, and borax contents of the gammons after storage are given in Table X. The salt content is excessive for satisfactory flavour quality. Depending on gammon size, dry-salting introduced from 50 to 100% more sodium chloride than the hardest cures described in Part I. Moreover, it was accompanied by excessive drying of the meat. The total borax content differed only slightly from that obtained when applied alone to the surface (cf. Table V).

The condition of the fat, as assessed by the peroxide oxygen content, was regarded as satisfactory (Table XI).

The numbers of large and pin-point bacterial colonies after storage (Table XI) were much the same as found after 32 days' storage of the hard, tank-cured product studied in Part I (cf. Table VII).

Packing bacon in a mixture of dry salt and borax would appear to have some preservative effect, but it is detrimental to flavour quality. The dry-salt pack not only introduces excessive quantities of sodium chloride but also causes undesirable drying.

IV. Dry Salting of Wiltshire Sides in Bales PROCEDURE

In the previous study it was observed that a high salt concentration in the meat retarded spoilage, but would be detrimental to flavour quality. The object of the present experiment was to ascertain if a high surface and lower internal salt concentration would sufficiently protect bacon from spoilage, and at the same time give a more palatable product.

The right and left sides of two hogs were used for this purpose. Each side was pumped with 19 stitches, but only half the usual stroke, the minimum pumping treatment considered to be practical. One side from each hog was wiped, and the blade pocket and hollow of the ribs filled with salt. Two sides were placed with meat surfaces together so that all the intervening space was filled with salt. After rubbing the skin surface of the sides with salt, they were baled and stored at 15.6° C. for 32 days. The remaining two sides were treated in the same manner except that they were cured for two days in a commercial tank prior to dry salting.

The quality of the sides after storage was assessed by organoleptic examination; determination of the chloride, nitrate, nitrite, and moisture contents of the gammons and backs, and salt distribution in the gammons; and by surface bacterial counts on the backs and deep-meat counts on the gammons. The analytical methods and sampling procedures were the same as described previously except that the surface counts were determined by diluting with both 4 and 10% brine and plating on agars of similar salt concentrations.

RESULTS

The changes in weight of the sides during cure and storage are of some interest. The increase on pumping was uniform and about half the usual amount of 5%. The average, over-all loss in weight of the dry salted sides was 9.9%, and 8.6% for the sides that had been partially tank-cured.

All of the sides had slimy, putrid areas on the skin surface where there was little or no salt. The surface colour of the entirely dry cured sides was dull and brownish as a result of methaemoglobin formation, while that of the partially tank-cured sides was satisfactory. Mould and slime were evident on some sides where the salt had fallen off. The odour of the freshly-cut surface of the gammons of the partially tank-cured sides was somewhat sour and of those entirely dry-salted, definitely off.

The deep-meat bacterial counts were variable, but all showed a considerable number of pin-point colonies (Table XII). There were usually more surface bacteria on the tank-cured than on the dry-salted sides. However, in all

TABLE XII

NUMBER OF BACTERIA ON THE SURFACE AND IN THE DEEP MEAT OF WILTSHIRE SIDES, DRY SALTED IN BALES FOR 32 DAYS AT 15.6° C.

Cure	Side No.	Surface organisms, log ₁₀ no. per sq. cm. as determined on agar containing:		Deep-meat organisms, log ₁₀ no. per gm. on 4% salt agar	
		4% Salt	10% Salt	Large colonies	Pin-point colonies
Tank + dry salt	1 R	6.33	5.63	3.51	7.30
	2 L	4.65	5.16	2.66	7.42
Dry salt	1 L	3.72	3.86	3.96	6.15
	2 R	4.73	4.62	2.70	7.58

instances the number of these organisms was considerably below the level at which slime is usually observed.

The results of the chemical analyses are given in Table XIII. The content of sodium chloride was relatively high in all sides. The tank-cured sides contained somewhat more nitrite, but the differences were small and probably would not account for the variations in keeping quality observed between the two types of cures. The difference in chloride content between the interior and surface of the gammons, amounting to about only 2%, was smaller than

TABLE XIII

CONTENT AND DISTRIBUTION OF CURING SALTS AND MOISTURE IN WILTSHIRE SIDES, DRY SALTED IN BALES FOR 32 DAYS AT 15.6° C.

Cure	Side No.	Position of sample ¹	Sodium chloride, %	Sodium nitrate, %	Sodium nitrite, p.p.m.	Moisture, %
Tank + dry salt	1 R	Inside	9.70	0.14	46	67.6
		Outside	11.83	0.06	14	58.0
		Gammon	10.65	0.08	10	60.6
		Back	4.90	0.01	3	62.1
	2 L	Inside	5.58	0.05	6	72.1
		Outside	7.48	0.03	63	66.8
		Gammon	6.22	0.03	6	67.1
Dry salt	1 L	Back	6.57	0.04	7	61.2
		Inside	8.18	0.08	5	71.0
		Outside	9.41	0.04	10	62.6
		Gammon	8.91	0.02	3	64.2
	2 R	Back	6.32	0.02	2	61.6
		Inside	6.34	0.05	5	69.9
		Outside	8.34	0.07	6	64.8
		Gammon	7.06	0.02	3	65.4
		Back	7.06	0.01	1	58.4

¹ See text.

expected. However, in spite of this, it would appear that the maintenance of a high salt concentration at the surface alone is not sufficient to prevent internal spoilage of Wiltshire bacon.

V. Effect of Surface Bacterial Contamination on Internal Spoilage

Three experiments were made to ascertain if internal spoilage in bacon was related to surface bacterial growth and the subsequent diffusion of elaborated enzymes into the meat.

Experiment 1

In the first experiment the relative effectiveness of salt contents of approximately 4 and 10% in retarding spoilage in and on the surface of bacon was studied. For this purpose the longissimus dorsi muscle was removed from a Wiltshire-cured back, and cut into four pieces of equal length. The two central portions were injected with 10 ml. of a saturated brine, and immersed in a brine of similar concentration for 72 hr. at 4.4° C. (40° F.). To simulate the interior of a side, one piece of high and another of low salt content were placed in the bottom of a vessel and covered with melted vegetable shortening. After the fat had hardened the remaining two pieces were placed on top and fat added until they were half covered. The vessel was covered and stored for 28 days at 15.6° C. In this manner bacon of both high and low salt content was exposed to, and excluded from, air.

Organoleptic examination of the surface samples after 14 days' storage showed that of high salt content to be in good condition, and the other to be fairly slimy with some mould growth, but no off-odours. At the end of the storage period, the exposed surfaces of the samples, which had been partially immersed in the fat, had off-odours and were covered with bacteria and some moulds. In contrast, the samples covered with fat during storage had no visible growth on the surface, but the piece containing the lesser amount of chloride had a sour odour.

The chloride and nitrite content of each sample was determined by methods described previously. The average difference between samples of low and high salt content was somewhat larger than expected (Table XIV). The nitrite content was highest in the samples that had been partially exposed to the atmosphere.

Samples for deep-meat bacterial counts were taken from the centre of each piece. The plates were incubated under both aerobic and anaerobic conditions. The most striking observation was the absence of pin-point colonies in meat containing 12 to 13% salt (Table XIV). Moreover, this type of colony was more prevalent in the partially exposed pieces of meat than in those buried in fat.

It would appear that a salt concentration as high as 13% is not sufficient to prevent surface spoilage but has some effect on deterioration in the deep meat. Internal spoilage is not due to enzymes diffusing into the meat from the

TABLE XIV

EFFECT OF SALT CONTENT AND EXPOSURE TO AIR ON THE NITRITE CONTENT, AND DEEP-MEAT,
AEROBIC, AND ANAEROBIC BACTERIAL COUNTS OF BACON
STORED AT 15.6° C. FOR FOUR WEEKS

Sample No.	Treatment	Content of curing salts		Bacterial count, \log_{10} no. per gm.			
		NaCl, %	NaNO ₂ , p.p.m.	Aerobic		Anaerobic	
				Large colonies	Pin-point colonies	Large colonies	Pin-point colonies
1	Covered	3.13	5	2.04	5.88	1.95	5.78
4	Covered	12.84	71	2.40	1.23	2.23	0
3	Exposed	5.72	220	1.97	7.46	1.70	7.45
2	Exposed	13.26	134	3.68	0	2.92	0

surface since the buried piece containing 3% salt had no visible surface growth and yet was spoiled internally. This conclusion is based on the assumption that appreciable diffusion of enzymes into the meat would occur only if the surface bacterial content was very high.

Experiment 2

The interior of a ham is essentially anaerobic. Since many anaerobes are inhibited by 4% salt, it has been suggested that spoilage of bacon is due to the growth of organisms on the surface, with the subsequent diffusion of enzymes into the deeper tissues. If this hypothesis is correct, it might reasonably be assumed that deterioration would be retarded if the meat were stored under anaerobic conditions, provided that the surface flora were aerobic or of the same type as present in the deep meat.

A Wiltshire gammon was stored in a desiccator under anaerobic conditions, obtained with hydrogen and palladinized asbestos, for four weeks at 21° C. (70° F.).

After storage the surface condition of the meat was poorer than when treated similarly under aerobic conditions. The ham had a bright red colour and a putrid odour. There was slime over most of the surface and some blue discolouration of the fat. The interior of the ham was likewise spoiled.

Samples (15 sq. cm.), taken from the surface for bacterial counts were plated on both nutrient and 4% salt agar, and incubated aerobically at 19.9° C. (68° F.) and anaerobically at room temperature. Deep-meat aerobic and anaerobic counts were made on a slice from the centre of the ham. These were plated on 4% salt agar only.

The results are given in Table XV. The presence of pin-point colonies on the surface is of interest. These were not usually found by aerobic methods, but since they were evident also after anaerobic incubation they are undoubtedly present on the surface. Storage under anaerobic conditions apparently accelerated rather than retarded surface spoilage of bacon. Hence

TABLE XV

NUMBER OF BACTERIA ON THE SURFACE AND IN THE DEEP-MEAT OF A WILTSHIRE GAMMON STORED ANAEROBICALLY FOR FOUR WEEKS AT 21° C.

Method of incubation	Surface counts, \log_{10} no. per sq. cm.				Deep-meat counts, \log_{10} no. per gm.	
	Nutrient agar		4% Salt agar		4% Salt agar	
	Large colonies	Pin-point colonies	Large colonies	Pin-point colonies	Large colonies	Pin-point colonies
Aerobic	8.31	0	8.34	Few	6.39	0
Anaerobic	8.28	Few	8.52	7.91	6.47	7.67

surface spoilage is not directly responsible for deterioration in the meat. The surface flora must consist essentially of facultative anaerobes.

Experiment 3

The study was designed to determine the effect on keeping quality of varying the internal bacterial content of bacon, while restricting surface growth to the lowest level possible. For this purpose 18 hams from different hogs were vein pumped with six curing pickles differing with respect to both the number and type of bacteria. The pickles were prepared by dividing a quantity of commercial curing solution into six equal portions, and subsequently sterilizing five of these. Organisms from sides and from pumping pickles were grown on nutrient agar and on agars containing 4 and 10% salt. Emulsions of these organisms were made in 10% brine, and used for inoculating four of the sterile solutions to give pump pickles of low and high contents of slime and pickle organisms. Bacterial counts for the pickles are given in Table XVI.

After pumping, the hams were cured in a commercial tank for seven days. To reduce the number of bacteria on the surface they were painted with a

TABLE XVI

BACTERIAL COUNTS OF PUMP PICKLES INOCULATED WITH VARIOUS AMOUNTS OF SLIME AND PICKLE ORGANISMS

Pickle	Log ₁₀ no. of bacteria per ml. as determined on:		
	Nutrient agar	4% Salt agar	10% Salt agar
Sterile	0.00	0.00	0.00
Low slime ¹	—	—	—
High slime	6.37	7.06	6.81
Low pickle	2.93	3.31	3.29
High pickle	6.38	6.97	6.92
Regular	2.81	3.29	3.19

¹ Plates misplaced.

4% aqueous solution of formaldehyde. They were then dried for three hours at room temperature, wrapped, and stored at 15.6° C. for 28 days. The formaldehyde treatment was repeated at weekly intervals during storage.

At the end of storage, surface bacterial counts were made on one ham selected at random from the three available for each pumping treatment. Two slices of each ham were taken for determination of deep-meat bacterial counts, and the chloride and nitrite content. In addition the formaldehyde content of a slice of the meat at positions adjacent to the fat and surface was determined for four hams (5).

There was no slime on any of the hams after storage. The odour of the internal meat of all hams was slightly sour.

Data for the surface and deep-meat bacterial counts are given in Table XVII. The surface counts were low. The number of large colonies in the deep meat more or less paralleled the bacterial content of the pump pickles employed. Based on a 4% increase in weight on pumping, the number was usually lower than would be expected, and indicates that appreciable growth

TABLE XVII

BACTERIAL AND CHEMICAL ANALYSES OF WILTSHIRE GAMMONS INOCULATED WITH VARIOUS AMOUNTS AND TYPES OF BACTERIA AND STORED AT 15.6° C. FOR FOUR WEEKS

Pickle	Sample No.	Bacterial counts			Sodium chloride, %	Sodium nitrite, p.p.m.	Formaldehyde, %			
		Surface, \log_{10} no. per sq. cm.	Deep-meat, \log_{10} no. per gm.				Inside ¹	Outside		
			Large colonies	Pin-point colonies						
Sterile	1	2.16	2.76	5.71	6.26	52	0.07	0.09		
	2	—	0.95	5.65	4.53	76	—	—		
	4	—	1.00	6.70	5.73	41	—	—		
Low slime	5	—	2.70	6.64	7.82	43	0.08	0.10		
	6	—	2.23	4.97	5.98	48	—	—		
	7	2.18	3.03	6.58	6.43	29	—	—		
High slime	9	3.30	5.33	6.35	5.52	24	—	—		
	11	—	5.45	7.51	6.07	136	—	—		
	17	—	4.31	6.39	6.63	126	—	—		
Low pickle	3	—	2.29	5.26	7.70	134	—	—		
	8	—	1.26	7.26	6.08	45	—	—		
	16	2.58	1.48	6.51	5.70	46	—	—		
High pickle	12	—	3.40	7.34	7.02	276	0.12	0.17		
	13	1.83	5.78	7.48	6.73	86	—	—		
	10	—	4.18	7.56	5.48	91	—	—		
Regular	14	—	1.64	6.18	6.78	65	—	—		
	15	1.60	1.48	6.41	5.78	76	—	—		
	18	—	1.15	6.40	6.38	41	0.13	0.15		

¹ See text.

did not occur under the conditions of the experiment. The number of deep-meat, pin-point colonies was in all instances high, and especially in hams pumped with the brine that had been heavily inoculated with pickle organisms.

The results of the chemical analyses are shown in Table XVII. There was some indication that the nitrite content varied directly with that of bacteria. The content of about 0.1% formaldehyde in the meat adjacent to the fat is surprisingly high since it was considered that little diffusion would occur under the experimental conditions employed.

Although bacterial growth on the surface was prevented, spoilage occurred in the interior of the hams. However, the extent of deterioration was less than normally occurs. This was presumably due in part to the presence of appreciable quantities of formaldehyde in the meat.

Acknowledgments

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THE BACTERIAL FLORA OF LOW-ACID VEGETABLES CANNED AT 212° F.

I. A PRELIMINARY STUDY OF THE EFFECTS OF VARIOUS PROCESSING PROCEDURES¹

By E. H. GARRARD², J. H. L. TRUSCOTT³, AND J. W. CONNER⁴

Abstract

Low-acid vegetables were processed in a water-bath at 212° F. for one-half to three hours continuously and also intermittently, as in home canning. Peas, corn, and snap beans processed by these methods showed the presence of many types of surviving aerobic and anaerobic bacteria, but none showed spoilage when containers were effectively sealed. The same was found true for commercially canned products. The addition of 50% tomato juice to snap beans considerably reduced the number of bacterial survivors and made possible the greatest number of sterile containers, even with processing time of one and one-half hours.

Introduction

War conditions of short supply of commercially canned low-acid vegetables such as peas, beans, asparagus, and corn, together with the encouragement given to home gardening, with consequent raw supplies of these products, have re-opened the question of the risks involved in the home canning of vegetables. Despite considerable publicity by radio and in print, there is not always agreement as to the risk involved. Commercial canning practices can be duplicated by the housewife with the use of a pressure cooker, but pressure cookers have not been available during the war period and even in normal times they are seldom used for home canning. Two methods are in general use, namely: immersion of the bottles or cans in a boiling water-bath, or the use of oven heat. In both instances the temperature reached in the food is approximately 212° F. Available canning recipe books do not agree closely as to the time required for the processing of low-acid vegetables, but generally it is advocated that they be held at 212° F. for two to three hours. In all probability only a small percentage of home canned food is processed for that length of time and moreover, the evidence on which the advocated time of processing is based is generally not available.

Such was the practical problem on which work was begun in the Horticultural Department in 1940. The objective was to use various advocated and new methods of processing low-acid vegetables in a boiling water-bath at 212° F. and if possible to select a method that seemed to have advantages over other methods. Early in the work it was found that spoilage occurred too seldom and usually after considerable storage periods. It was therefore decided to make microbiological assays, and this preliminary paper is a

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condensation of the first year's study of the microbiology of the finished products**.

Methods of Processing

One obvious, variable factor that can materially affect the efficiency of a processing method in the canning of low-acid vegetables is the cleanliness of the product when packed. Some of the earlier materials were cleaned and handled in a manner too aseptic to be considered typical of kitchen conditions. Thereafter, the cleaning, trimming, and cutting of the vegetables were done by girls with no laboratory training in order to emulate household procedures. The water used for washing and covering solutions was from the College mains and occasionally contained chlorine. All vegetables used were fresh and free from obvious diseases.

Most household canning recipes advocate the "sterilization" of all utensils and containers in boiling water. Often the equipment is not completely immersed, nor is the water boiling, and the amount of heat available in the average kitchen is usually inadequate to keep at the boiling point the amount of water necessary to completely immerse the equipment. Consequently, the following method was used. All utensils were thoroughly washed in warm, soapy water using brushes or cloths to rub all surfaces. They were then rinsed under the tap and placed in a bath containing approximately one tablespoon of chloride of lime to each gallon of water, from which they were taken and allowed to stand wet on a washed table. Just before use they were again rinsed under the tap until the odour of chlorine had disappeared. Such a method of cleaning utensils is considered as effective and more practical than those usually advocated.

In these initial experiments, asparagus, carrots, peas, and snap beans were processed. Unfortunately, owing to the lateness of the season when the project was started, a very small pack of asparagus was put up and the bacteriological results were too inconclusive to merit reporting. Also, only a small crop of peas was available, with the result that only 40 containers were analysed. Most of the canning that summer was done with snap beans.

All the vegetables were blanched in boiling water for periods up to four minutes, according to commercial practice, and cooled in tap water. They were then packed in clean containers and various covering solutions added. Usually, it was a 2% common salt solution added at room temperature.

Sixteen ounce household "sealers" were used in addition to No. 2 cans as containers. In all instances they were completely immersed in the water-bath and the processing time was measured from the time when the centre temperature of the packed container reached 210° to 212° F. Partial vacuums were obtained in cans either by preheating the open can to a temperature of about 180° F. in a shallow water-bath or by punching a hole $\frac{3}{64}$ in. in diameter in the top before immersion in the warmed processing

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bath. As soon as processing was finished the hole was immediately soldered over. Glass containers are automatically exhausted after partial tightening of screw tops or by the lifting of spring top closures. The degree of vacuum obtained in common types of glass containers varies since it depends on when the seal takes effect during the cooling process. Considerable spoilage was experienced from glass containers that did not seal. Cans only were used in later work, partly because they could be quickly cooled in cold water at the end of the processing period.

Processing times at 212° F. used with various lots of vegetables were as follows:— one-half, one, one and one-half, two, two and one-half, and three hours; and one-half, one, and two hours repeated on three successive days.

Bacteriological Methods

Because of the number of processing methods involved in this preliminary survey, it was necessary to limit the scope of the bacteriological analyses. Culture media used were those advocated by Cameron (1). These consisted of (a) dextrose tryptone agar for the isolation of aerobic, flat sour thermophiles and facultative anaerobes; (b) liver broth for thermophiles and putrefactive mesophilic anaerobes; and (c) sulphite agar for thermophilic anaerobes producing hydrogen sulphide. His media were augmented with grass medium for anaerobic bacteria devised by Castell (2). Plates, tubes, etc. were incubated for 7 to 10 days at temperatures of 32° C. and 55° C. for mesophilic and thermophilic types respectively.

Preliminary experiments with various vegetables had shown that plate counts from samples examined immediately after processing showed little or no correlation with processing times. Consequently, data are reported only on the presence of bacterial growth and results are expressed in percentage of containers showing various types of bacterial survivors.

The finished products were examined immediately after processing and cooling, or if some delay ensued, they were stored at 32° F. Duplicate containers were incubated for 7 to 10 days at 32° C. or 55° C. before sampling, and others to be examined later were held at 65° F. for periods up to 12 months. Before sampling, the containers were carefully examined for imperfections. They were then washed with soap and water, wiped with alcohol, and flamed. Methods used for opening the containers, sampling, etc. were those advocated by Tanner (4).

Results

Peas

Lots of peas were processed for two hours at 212° F. and others intermittently for one-half hour periods on three successive days and held at room temperature between heatings. Among the containers picked at random from those processed only one spoiled after nine days' incubation at 32° C. and it was found to be ineffectively sealed. The percentage of containers showing various surviving types of organisms are shown in Fig. 1.

All containers of peas sampled were positive to some form of bacterial growth. However, it must be emphasized that none showed spoilage when effectively sealed. The intermittent method as compared to the two hour continuous process resulted in fewer containers showing survivors and a marked decrease in those containing aerobic thermophiles.

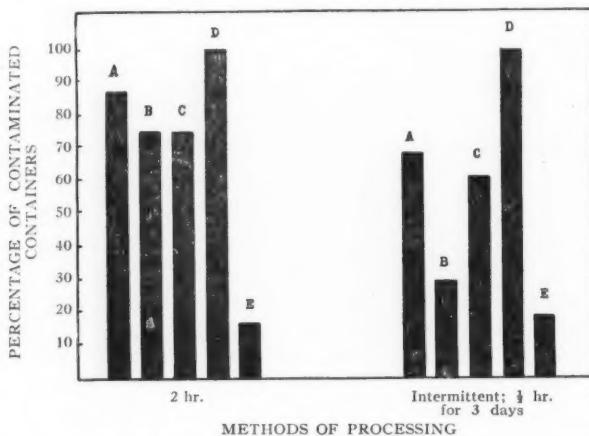


FIG. 1. Percentage of containers of peas showing bacterial survivors after two hours' heating and intermittent heating at 212° F. A—*aerobes* at 32° C.; B—*aerobic thermophiles* at 55° C.; C—*anaerobes* at 32° C.; D—*anaerobic thermophiles* at 55° C.; E—*hydrogen-sulphide-producing anaerobes*.

Carrots

Sixty-one containers of diced carrots were examined, but owing to lack of incubator space at the time, no samples were examined for the presence of thermophiles and the results shown give only aerobic and anaerobic growth after incubation at 32° C. for 10 days. None showed spoilage. The containers were chosen from carrots processed for one and one-half, two, and three hours at 212° F. Results are shown in Fig. 2. According to these results, the increased processing time seemed to exercise little influence in reducing the percentage of containers containing bacterial survivors at 32° C. However, a greater number of samples and the inclusion of isolation of thermophilic types might have presented a different picture. As shown, the reduction in the number of containers showing bacterial survivors at 32° C. indicated better results than those obtained with peas.

Snap Beans

Forty containers of processed snap beans were chosen for analysis. As with carrots, there was little to choose between one and one-half and two hours' heating with regard to the number of containers showing survivors and they are included in one group in Fig. 3. In some instances the number was reduced with three-hour heating. As with peas, intermittent heating was more effective against thermophiles.

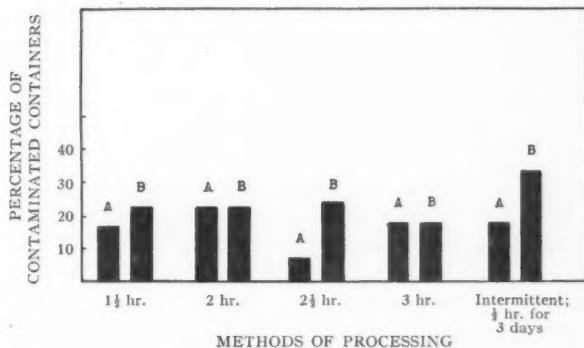


FIG. 2. Percentage of containers of carrots showing bacterial survivors at 32° C. after one and one-half, two, two and one-half, and three hours' heating and intermittent heating at 212° F. A— aerobes at 32° C.; B— anaerobes at 32° C.

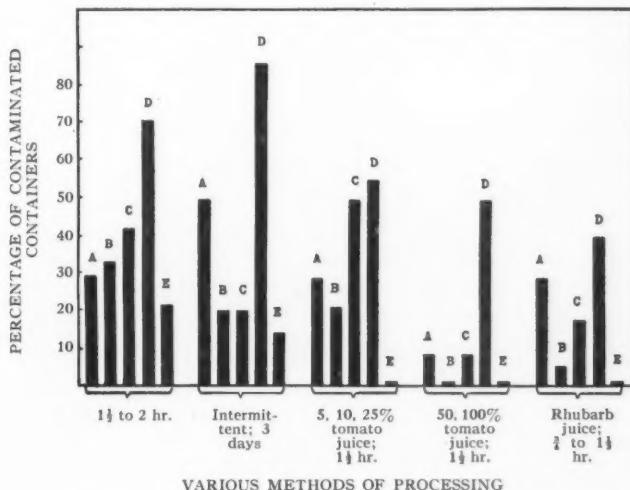


FIG. 3. Percentage of containers of beans with and without tomato and rhubarb juice showing bacterial survivors after various periods of heating at 212° F. A— aerobes at 32° C.; B— aerobic thermophiles at 44° C.; C— anaerobes at 32° C.; D— anaerobic thermophiles at 55° C.; E— hydrogen-sulphide-producing anaerobes.

Addition of Various Acids

Since one main difference between the acid and low-acid horticultural products is their pH, various acids were added before processing in such proportions that the pH of the products approximated that of tomatoes. (Tomatoes are canned commercially in a boiling water-bath; their pH varies between 4.3 and 4.8.) Most of the acids used rendered the products unpalatable and no microbiological assays were made when palatability was unsatisfactory.

factory. Citric and acetic acids are sometimes advocated as additions to non-acid vegetables, but both were discarded after the flavour of the finished product was tested.

It was decided to add tomato juice in varying concentrations to snap beans in order to determine the effects of the juice on the microbial flora, as well as to increase the ascorbic acid content. Previous tests had determined that a 50% tomato juice mixture, with or without 2% common salt, produced a palatable flavour blend with peas, snap beans, asparagus, and corn. The juice, fresh or canned, was diluted with water and used as a covering solution on the vegetables packed in the containers. The concentrations of tomato juice used with snap beans varied from 5 to 100% and all containers were heated for one and one-half hours.

Electrometric determination showed that, after processing, the pH of the contents of the lots with 5, 10, and 25% tomato juice varied between 5.85 and 6.0; the pH in the lots containing 50 to 100% varied between 4.5 and 4.8. Low-acid vegetables such as beans and asparagus generally show pH values between 5.0 and 6.5 with most of them higher than 5.5, hence the addition of 50 to 100% tomato juice placed the beans in the typically acid range of tomatoes.

Rhubarb juice was added to another series. With amounts varying from 20 to 60 ml. per 16 oz. jar, pH values of the finished products after processing ranged from 4.1 to 5.3.

Results

On the basis of containers showing survivors there was little difference with additions up to 25% or with additions between 50 to 100% tomato juice; but the addition of 50% or more juice produced a marked reduction in the number of containers showing survivors of all types of organisms except thermophiles. These two groups were chosen for comparison with beans heated without tomato juice.

Eighty containers of processed snap beans covered with tomato juice were analysed. None showed spoilage. In the group containing from 5 to 25% tomato juice there was a lower percentage of containers showing survivors of the various groups of bacteria than in beans processed without tomato juice, but the difference was hardly sufficient to justify continuing with these concentrations. However, in this group four containers showed no bacterial survivors. More promising results were obtained with 50 to 100% concentrations of tomato juice (Fig. 3). The percentage of containers showing various types of survivors, with the exception of anaerobic thermophiles, was considerably lowered. The percentage of containers showing survivors were fewer (with the exception of aerobes at 32° C.) than in commercially packed food as shown in Fig. 4. Nine containers were negative to any form of bacterial growth.

The addition of rhubarb juice, despite the lower pH, gave less satisfactory results than did 50 to 100% tomato juice. Of particular interest in all these

experiments is the high percentage of containers showing surviving anaerobic thermophiles. Their presence bears out the recent findings of Castell (3) who found high counts of such organisms in many foods and food ingredients.

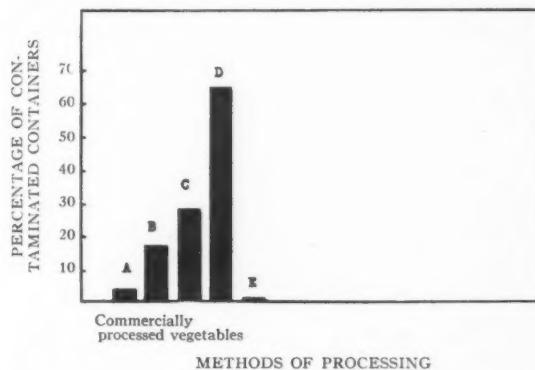


FIG. 4. Percentage of containers of commercially processed vegetables showing bacterial survivors. A—*aerobes* at 32° C.; B—*aerobic thermophiles* at 55° C.; C—*anaerobes* at 32° C.; D—*anaerobic thermophiles* at 55° C.; E—*hydrogen-sulphide-producing anaerobes*.

Examination of Commercially Canned Products

As so many containers of vegetables processed at 212° F. had shown bacterial survivors, it was thought necessary to obtain and analyse commercially canned foods for comparison. Forty-six cans representing several brands of peas, beans, corn, carrots, spinach, and pumpkin were obtained from retail stores. All cans were normal externally and were incubated at 32° C. or 55° C. before being examined.

Thirteen of the cans did not exhibit any signs of bacterial growth. Anaerobic thermophiles were present in 67% of the cans. On the other hand, only 4.3% showed the presence of aerobic types at 32° C. It is apparent from these results that even commercially processed products may possess many types of surviving bacteria and yet show no signs of spoilage. Fig. 4 shows the percentage of containers with bacterial survivors.

Discussion

It is apparent from this preliminary survey that heating of low-acid vegetables for periods up to and longer than two hours at 212° F. leaves many surviving types of organisms, which although present, do not necessarily cause spoilage. Probably, no practical method of processing can guarantee sterility at 212° F. Consequently, these results do not alter the premise that heating at 212° F. is attended with risk. On the other hand, it was shown that even commercially packed vegetables commonly contain considerable numbers of live bacteria.

The data, as presented, indicated that very favourable and encouraging results were obtained by introducing tomato juice as a means of checking or preventing bacterial growth. While it is acknowledged that this initial work is hardly extensive enough to justify the wholesale use of tomato juice in vegetable preservation, it does open a field of investigation that shows considerable promise. These and subsequent experiments with 50% concentrations of tomato juice have led to work in which other phases of bacteriological analysis are being undertaken.

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DRIED WHOLE EGG POWDER

XVII. OBJECTIVE TESTS AND BAKING QUALITY¹

BY MARGARET REID² AND JESSE A. PEARCE²

Abstract

Potassium chloride value, refractometric value, the fluorescence of defatted egg powder in 10% potassium chloride, and the fluorescence of untreated dried material correlated equally well with the foaming volume of a mixture of dried egg, sugar, and water, and with the loaf volume of the sponge cake prepared from the powder (r) values from .81 to .90). A significantly closer relationship was observed between foaming volume and loaf volume ($r = .96$). Since foaming volume was more precise and easier to determine than baking volume, it was concluded that foaming volume was a more desirable test of baking quality.

Introduction

Previous work in these laboratories on objective methods for measuring the quality of egg powders (5) has been confined to assessing their palatability. Since the use of dried egg for baking is as important as its use in the preparation of egg dishes and since the estimation of baking quality by loaf volume measurements is cumbersome, it was considered advisable to study the relationship of some of these objective tests to baking quality.

Materials and Methods

Nine powders from the main chamber and secondary collectors of plants currently producing dried egg in Canada and four powders prepared in a laboratory spray drier (8) were used in this experiment. The powders ranged in quality from excellent to inedible.

The objective tests used included: solubility in 10% potassium chloride (5), the refractive index of the defatted material in 5% sodium chloride (6), the fluorescence of the defatted powder in 10% potassium chloride (4), the fluorescence of untreated dried egg (2), and foaming volume (1). In addition an index of soluble protein materials was obtained by evaluating the nitrogenous material in the filtrate used to determine potassium chloride solubility (Kjeldahl). This was recorded as the percentage of protein dissolved, calculated on the weight of sample taken. Results from the above tests were compared with volumes of sponge cakes (7) as the criterion of baking quality.

Procedures for determining foaming volume and loaf volume varied somewhat from those previously described. Duplicate determinations of foaming volume were made using 19 gm. of egg powder, 60 gm. of sugar, and 56 ml. of distilled water. The sugar and egg powder were mixed and the water added gradually to form a homogeneous mixture. This mixture, after being warmed

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to 40° C. (104° F.) in a water-bath, was removed to a cabinet, regulated at 40° C. and a relative humidity of 80%, and beaten for 10 min. in a 'Mixmaster' set at number 10 speed. The resulting foam was then transferred to a graduate and its volume measured.

Ingredients for loaf volume determinations were 12 gm. of dried egg, 40 gm. of sucrose, 40 gm. of unsifted flour, and 38 ml. of distilled water. After blending the sugar and egg in a bowl, 5 ml. of water was added to make a paste, which was then formed into a homogeneous fluid with the remainder of the water. This mix was allowed to stand in the high humidity cabinet for 30 min., then removed to the baking laboratory and beaten for 10 min. at number 10 speed on a Mixmaster under laboratory conditions of 27° C. (80° F.) and 65% relative humidity. The flour was added with the least possible mixing otherwise the ultimate loaf volume was considerably reduced. The batter was transferred rapidly to a baking tin ($10 \times 5 \times 5$ cm.) and baked for 40 min. at 170° C. (338° F.). Four cakes were made from each sample of powder and the volumes measured (3).

Results

The correlations between objective tests and loaf volume are given in Table I and shown graphically in Figs. 1 and 2. All correlations were highly significant. The soluble nitrogen was the least satisfactory of the measure-

TABLE I
CORRELATIONS OF QUALITY TESTS WITH FOAMING VOLUME AND LOAF VOLUME

Quality test	Correlation with	
	Foaming volume	Loaf volume
Soluble protein index	.79**	.78**
Potassium chloride value	.90**	.88**
Refractometric value	.85**	.89**
Fluorescence of potassium chloride extract	-.88**	-.85**
Fluorescence of powder	-.87**	-.81**
Foaming value	—	.96**

**Indicates 1% level of statistical significance.

ments for predicting baking quality. The potassium chloride value, refractometric value, the fluorescence of a 10% potassium chloride extract of defatted egg powder, and the fluorescence of untreated dried egg were more satisfactory and about equally effective as methods of predicting loaf volume. Foaming volume was the most satisfactory method for predicting baking quality ($r = .96$). This correlation coefficient was significantly higher than all other coefficients obtained.

In spite of the high correlation, foaming volume could be used to predict loaf volume only within ± 30 ml. However, this lack of accuracy may be

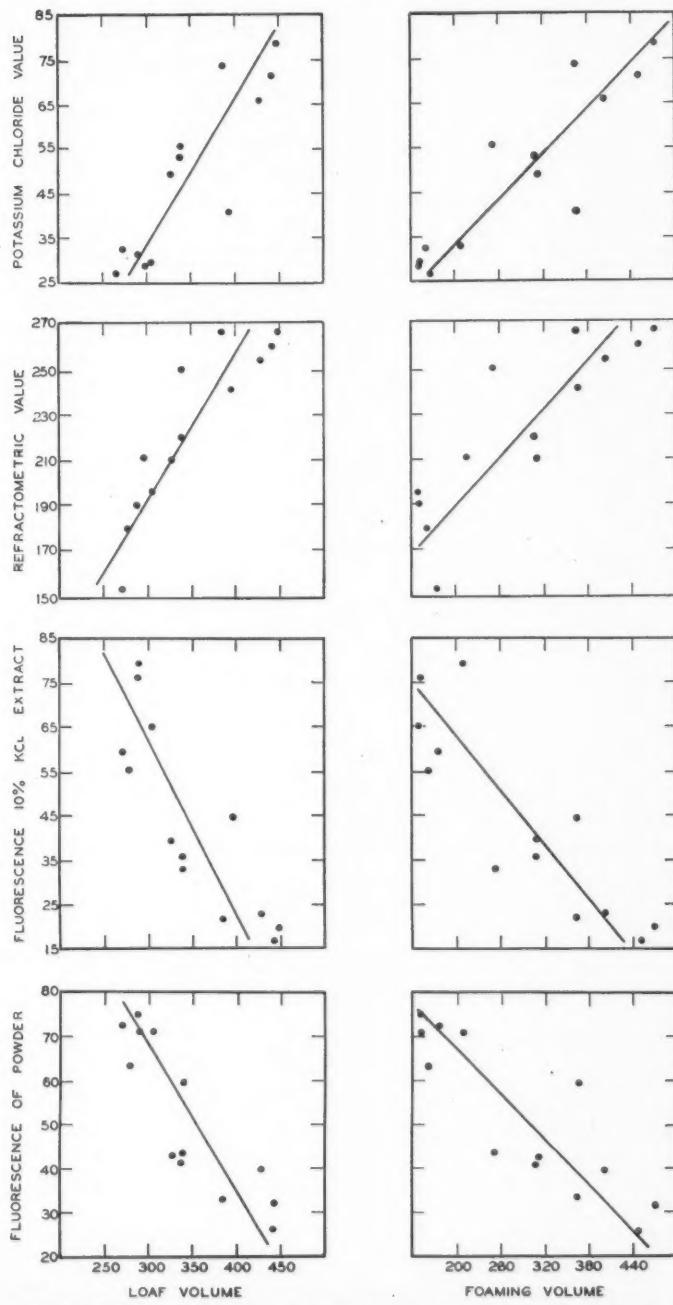


FIG. 1. Relation of objective tests to foaming and loaf volume measurements.

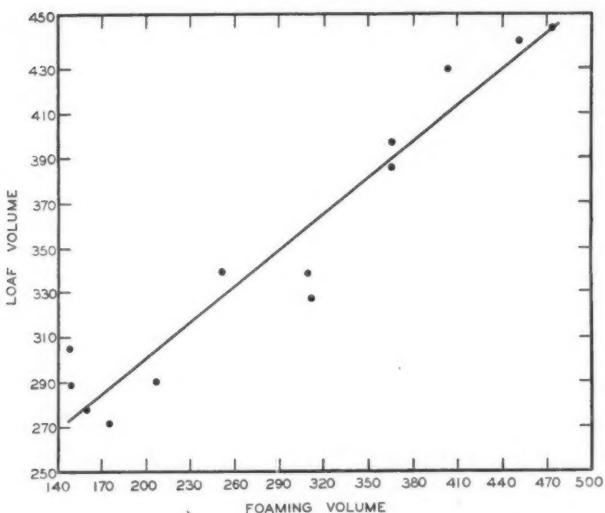


FIG. 2. Relation between foaming volume and loaf volume measurements of dried egg.

attributable to the large experimental error in determining loaf volume (standard deviation for replicate loaves 30 ml.; 10% of the mean). Foaming volume was measured with much greater accuracy (standard deviation, 6 ml.; 2% of the mean). In addition to these considerations, foaming volume was more practical for laboratory use; less time and less complicated apparatus were required for making a determination. Therefore it was concluded that foaming volume was the most satisfactory of the methods used in this study for evaluating the baking quality of dried whole egg powder.

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**A SURVEY OF THE VITAMINS A AND D POTENCIES OF
THE LIVER OIL OF ATLANTIC COD
(*GADUS MORRHUA* L.)¹.**

BY L. I. PUGSLEY², C. A. MORRELL³, AND J. T. KELLY⁴

Abstract

A survey has been made of the variations and of some of the factors influencing the variations of the vitamins A and D potencies of the liver oil of cod landed at ports in Nova Scotia, New Brunswick, and the Gaspé peninsula of Quebec. An increase in the vitamin A potency was paralleled by an increase in the vitamin D potency and the oil content of the liver increased with the percentage liver in the fish. An increase in the oil content of the liver and of the liver content of the fish was accompanied by a decrease in the concentration of vitamins A and D in the oil. The vitamin potency of the oil tended to decrease as the fishing season advanced from June to October and the oil content of the liver increased during this period. When the yield of vitamins was expressed per 100 gm. of fish there was no apparent seasonal change in potency indicating that the seasonal changes observed were due to dilution. A relationship was observed between the stages in the spawning cycle and the oil content of the liver. Fish classed as "steaks" (six to eight years) yielded a liver oil higher in vitamins A and D potencies than "market cod" (four to six years) and the liver oil of "scrod" (three to four years) had the lowest vitamins A and D potencies.

The commercial production of an oil for medicinal and veterinary use from the livers of Atlantic cod (*Gadus morrhua* L.) frequently referred to by the more recent synonym (*Gadus callarias* L.) landed at ports in the Maritime provinces of Canada has shown a marked increase during the last four years. From data compiled by the Oils and Fats Administrator (10) of the Wartime Prices and Trade Board (Table I), it is seen that the production of common cod oil, often described as sun rotted oil, has shown a marked decrease and the production of refined cod liver oil has increased considerably.

In view of the importance of this product to our national economy and the meagre data available on the vitamins A and D potencies of authentic samples

TABLE I
THE PRODUCTION OF COD LIVER OIL IN CANADA

Year	Common cod oil, gal.	Crude cod liver oil, gal.	Refined cod liver oil, gal.
1941	74,489	215,269	94,158
1942	45,607	203,561	127,905
1943	27,528	254,529	168,099

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of cod liver oil it was deemed advisable to obtain information on the product as landed at ports in Nova Scotia, New Brunswick, and the Gaspé region of Quebec. The main objects of the investigation were to obtain data on factors influencing the vitamins A and D potencies of the oil that would be of value for the guidance of fishermen and operators of processing plants.

Collection of Samples

The collection of the livers was supervised by the Inspectors of the Department of Fisheries and by members of the scientific staff of the Fisheries Research Board of Canada at the Atlantic Fisheries Experimental Station, Halifax, N.S., and the Gaspé Fisheries Experimental Station, Grand River, Que. The following places were selected for collecting the livers: Lockeport, N.S., approximately 150 miles from Halifax; North Sydney, N.S., on Cape Breton Island; Petit Rocher, N.B., on Chaleur Bay approximately 12 miles from Bathurst; and Grand River, Que., on the Gaspé peninsula.

It was planned to collect monthly samples of livers from each of the above places during the fishing season but factors such as weather and fishing conditions beyond control interfered, and only at Petit Rocher and Grand River was the original sampling program completed. At Grand River, samples were collected from fish caught 1 to 2 miles from shore and these were termed 'inshore fish'; at the same time samples were taken from the Miscou fishing banks 30 to 40 miles from shore and these were termed 'off-shore fish'. The fish from Lockeport, North Sydney, and Petit Rocher represent fish caught within 5 to 20 miles from these places. In addition to the above samples, livers were obtained from fish of different weight groups, corresponding to approximately the following age groups, scrod three to four years, market cod four to six years, and steaks six to eight years. These were obtained from fish landed at Canso, Halifax, and Mulgrave in Nova Scotia.

Methods

The whole fish were landed by the fishermen and weighed, the livers removed in the presence of one of the Fisheries Inspectors and the sex and stage of maturity noted. The samples consisted of 70 to 75 fish taken at random. The livers from these were placed in cans and stored in a refrigerator at -10° C. for four to six days when they were removed, packed in boxes containing wood shavings and carbon dioxide snow, and shipped to Ottawa. On arrival the livers were minced in the frozen condition and thoroughly mixed. Duplicate 10 gm. samples of liver were taken and the vitamin A and oil content were determined using chloroform extraction and the antimony trichloride reaction for vitamin A as described by Pugsley (7).

The remainder of the minced sample was placed in covered cans and heated in an autoclave for one hour at 12 to 15 lb. steam pressure. The oil was obtained from the cooked material by decantation and by centrifuging off the oil from the residue. The oil was dried by adding anhydrous sodium sulphate, filtered and stored at -4° C. in full brown bottles. Vitamin D

assays were carried out on these samples essentially by the method outlined in the British Pharmacopoeia (2, pp. 597-600) using the International Standard as a reference standard. The assays were done at three dosage levels each for the standard and sample with at least 10 rats per dosage level and the potency and limits of error were calculated according to Bliss and Marks (1). Where the limits of error exceeded $\pm 35\%$ with p at 0.05 the assays were repeated and the combined result reported.

Results

The date and place of catching, the mean weight of the fish in the sample, the percentage of liver in the fish, the percentage of oil in the liver, and the vitamins A and D potencies of the oil are shown in Table II. The samples from each locality are arranged chronologically to show the seasonal variations. It is seen from the mean weight and the standard deviation of the individual

TABLE II

DATE AND PLACE OF CATCHING, WEIGHT OF FISH, PERCENTAGE OF LIVER IN FISH, PERCENTAGE OF OIL IN LIVER, AND VITAMINS A AND D POTENCIES OF OIL

Date	Locality	Mean wt., kgm.	S.d.*	Liver in fish, %	Oil in liver, %	Vitamin A, L value per gm. oil	Vitamin D, I.U. per gm. oil		
							Mean	Range	Sm $\times 1.96$
1942									
9/6	— I**	Grand River, Que.	4.51	2.49	2.81	43.8	5700	152	99 231
9/6	— O		3.66	1.65	2.90	36.2	6600	66	51 87
13/7	— I		4.10	3.37	2.96	40.6	6000	55	39 77
13/7	— O		4.06	1.98	2.72	38.5	4900	56	40 80
12/8	— I		4.26	2.98	3.48	56.4	2300	42	32 55
12/8	— O		4.41	2.21	3.47	42.7	4400	44	32 60
17/9	— I		3.72	2.45	3.61	53.0	2400	22	18 29
17/9	— O		4.25	1.89	3.26	54.9	2300	40	29 54
17/10	— I		3.73	2.71	4.82	59.3	2300	32	22 45
17/10	— O		3.60	1.76	4.93	57.5	2200	27	23 33
1941									
11/7	Petit Rocher, N.B.	4.31	2.04	2.04	46.0	3600	91	62	134
26/7		4.53	2.55	2.55	46.6	5100	108	83	141
25/8		5.77	4.19	2.70	36.0	3600	83	58	119
24/9		4.50	1.84	2.63	54.4	4200	86	72	104
21/10		4.28	3.15	2.62	44.0	2100	85	66	110
8/7	North Sydney, N.S.	4.91	2.98	2.25	45.4	4500	145	104	201
1/8		4.07	3.36	3.75	53.5	3700	80	52	124
23/10		3.71	1.95	4.34	52.5	2300	78	51	120
13/6	Lockeport, N.S.	5.11	5.34	1.87	22.8	13600	306	231	404
25/7		5.50	4.18	2.83	34.6	7300	258	195	341
7/8		6.74	4.60	3.85	49.2	4500	212	150	299

* S.d. = Standard deviation.

** I = Inshore.

O = Offshore.

NOTE: For the purposes of this report L value per gm. is regarded as an approximation of the vitamin A potency in International Units.

weights that considerable variation occurred in the weight of the fish within each sample, and the fish from Lockeport were larger than the fish from the other places. With the exception of the samples from Petit Rocher there is a tendency for the percentage of liver in the fish to increase as the fishing season advances from June until October. This is accompanied by an increased yield of oil and a decrease in the potency of the oil in vitamins A and D. These results show that during the summer and fall months fat is stored in the livers of the fish and this causes a dilution of the oil with respect to vitamins A and D. There is not a significant difference between the values obtained for fish caught inshore and offshore. The vitamin D potency of the oil from the fish landed at Lockeport is higher than that from the other locations sampled.

To show that the actual concentration of vitamins A and D in the liver are independent of seasonal variations and that the changes observed are due to seasonal variations in the oil content of the liver, the yields of vitamins A and D per 100 gm. of fish were calculated. These are shown in Table III. The definite seasonal trend of the vitamins A and D potencies of the oil is no

TABLE III
THE VITAMINS A AND D POTENCIES PER 100 GM. FISH

Date	Locality	Vitamin A, L value per 100 gm. fish	Vitamin D, I.U. per 100 gm. fish
1942	Grand River, Que.	7000	187
9/6 — I*		6900	69
9/6 — O		7200	61
13/7 — I		5100	59
13/7 — O		4500	82
12/8 — I		6500	65
12/8 — O		4600	42
17/9 — I		4100	72
17/9 — O		6600	92
17/10 — I		6200	77
1941	Petit Rocher, N.B.	3400	85
11/7		6100	128
26/7		3500	81
25/8		6000	123
24/9		2400	98
21/10	North Sydney, N.S.	4600	148
8/7		7400	161
1/8		5200	178
23/10	Lockeport, N.S.	5800	130
13/6		7100	253
25/7		8500	401
7/8			

* I = Inshore.
O = Offshore.

longer apparent. However a regional difference in the yield of vitamin D is seen, e.g., the fish landed at Lockeport and North Sydney yield a larger amount of vitamin D per 100 gm. of fish than those from Petit Rocher and Grand River.

The correlation coefficients of the various relationships presented in Tables II and III were calculated and are shown in Table IV. From these results

TABLE IV

THE CORRELATION COEFFICIENTS OF THE RELATIONSHIPS PRESENTED IN TABLES II AND III

Relationship between	r^*	Remarks
Vitamins A and D potencies of oil	+ .776	Highly significant
Percentage oil in liver and vitamin D potency of oil	- .635	Highly significant
Percentage oil in liver and vitamin A potency of oil	- .827	Highly significant
Percentage liver in fish and vitamin D potency of oil	- .517	Significant
Percentage liver in fish and vitamin A potency of oil	- .550	Significant
Percentage liver in fish and percentage oil in liver	+ .688	Highly significant
Vitamins A and D potencies per 100 gm. fish	+ .518	Significant
Percentage oil in liver and vitamin D potency per 100 gm. fish	- .043	Not significant
Percentage oil in liver and vitamin A potency per 100 gm. fish	- .020	Not significant
Percentage liver in fish and vitamin D potency per 100 gm. fish	+ .073	Not significant
Percentage liver in fish and vitamin A potency per 100 gm. fish	+ .343	Not significant

* For 19 degrees of freedom r at 5% = .433, at 1% = .549.

the following relationships are shown to exist: (1) the vitamin A potency increases as the vitamin D potency increases both when the potencies are expressed per gm. of oil and per 100 gm. of fish; (2) the larger livers contain a higher proportion of oil; (3) the higher the proportion of oil in the liver and the greater the percentage of liver in the fish the less was the vitamin concentration in the liver oil. These results show that the seasonal changes in the oil content of the liver are responsible for the seasonal changes in the vitamins A and D potencies of the oils; in other words the changes in vitamin potency are due to dilution.

During the non-spawning period codfish are actively feeding and storing fat in the liver; this fat is utilized during the spawning period. Observations on the stage of development of the gonads expressed as the stage of maturity of the fish were recorded for the samples of fish collected at Petit Rocher and Grand River. The criterion used for defining the different stages of maturity are shown in Table V and the percentage of fish in each stage for the samples landed at Grand River and for three samples from Petit Rocher are recorded in Table VI. Similar information for the other samples was not available. It is seen from the data on the fish landed at Grand River that as the season advances from June to October the percentage of fish in Stages 3, 4, and 5 decreases and the percentage in Stages 1 and 2 increases. On the other hand this shift is not so apparent in the samples from Petit Rocher where there

TABLE V
DEFINITION OF STAGES OF MATURITY BASED ON THE CONDITION OF THE GONADS

	Female	Male
Stage		
1 Small and very firm		String-like, and slightly wavy
2 Firm		Whitish and more wavy
3 Reddish and granular		Whitish, wavy, and larger
4 Reddish to golden, opaque and larger with eggs attached		Whitish and distinctly wavy, curving almost in loops
5 Spawning stage. Eggs are transparent and running		Spawning stage. Milt running
6 Whitish, empty, and flabby		Shrunken and becoming string-like

TABLE VI
THE PERCENTAGE OF MALE AND FEMALE FISH AND THE PERCENTAGE OF FISH IN MATURITY STAGES 1 TO 6 IN EACH LOT OF FISH FROM GRAND RIVER AND PETIT ROCHER

Date	Locality	Male, %	Female, %	Stage of maturity					
				1	2	3	4	5	6
Percentage of fish in stage									
1942									
9/6 — I*	Grand River	21.3	78.7	12.0	52.0	16.0	14.7	5.3	0.0
9/6 — O		30.3	69.7	10.5	29.0	27.6	23.7	9.2	0.0
13/7 — I		54.8	45.2	25.6	30.5	20.7	17.1	3.7	2.4
13/7 — O		70.6	29.4	10.6	33.4	28.0	22.6	5.4	0.0
12/8 — I		42.0	58.0	40.6	54.1	4.0	0.0	0.0	1.3
12/8 — O		55.5	44.5	18.9	48.6	20.3	5.4	5.4	1.4
17/9 — I		48.0	52.0	21.4	70.6	6.7	1.3	0.0	0.0
17/9 — O		37.4	62.6	24.0	58.7	8.0	9.3	0.0	0.0
17/10 — I		55.5	44.5	22.2	59.7	16.7	1.4	0.0	0.0
17/10 — O		52.0	48.0	25.3	58.7	8.0	4.0	0.0	4.0
1941									
25/8	Petit Rocher	42.6	57.4	22.6	22.6	20.0	16.0	9.3	9.4
24/9		56.0	44.0	22.6	10.7	6.6	20.0	21.3	18.8
21/10		53.5	46.5	22.6	24.0	10.7	9.3	0.0	33.4

* I = Inshore.

O = Offshore.

is a more uniform distribution of the different stages within each sample. To determine the relationship between the stage of maturity and the yield of oil, the correlation coefficients were calculated for the samples landed at Grand River and the results are shown in Table VII. The positive correlation between the percentage of oil in the liver and the percentage of fish in Stages 1 and 2 shows that the yield of oil tends to increase with the increasing proportion of fish in the resting Stages 1 and 2 of the spawning cycle. On the other hand the negative correlation found for the prespawning Stages 3 and 4 indicates that these stages are associated with a low yield of oil.

TABLE VII
RELATION BETWEEN STAGE OF MATURITY AND YIELD OF OIL

Relation between % oil in liver and % of fish in:	<i>r</i> *	Remarks
Stage 1	+ .650	Significant
Stage 2	+ .845	Highly significant
Stage 3	- .869	Highly significant
Stage 4	- .867	Highly significant

* With 8 degrees of freedom *r* at 5% = .632, *r* at 1% = .765.

In order to show that the age of the fish is an important factor in determining the vitamins A and D potencies of the oil, samples of liver were collected from fish segregated into the different weight groups according to a marketing classification. These weight groups correspond to approximately the following age groups: scrod, three to four years; market cod, four to six years; and steaks, six to eight years. The results are shown in Table VIII. It is seen that the older fish (steaks, six to eight years) yield a liver oil of higher vitamins A and D potencies than the younger fish (market cod, four to six years, and scrod, three to four years). The effect is more significant with respect to vitamin D than with vitamin A. The sample of steaks and scrod landed on July 30 at Halifax appear to be out of line in their respective groups. Upon checking the information about these samples it was found there was the possibility of an error in labelling these cans of liver. When these two samples are interchanged the age effect is more pronounced.

Discussion

Data on the vitamins A and D potencies of authentic samples of oil from the livers of codfish landed at ports in the Maritime provinces are very meagre and consist chiefly of reports on analysis of isolated samples sold commercially. Since it is well known that samples of this nature often represent an oil blended with other fish oils and frequently fortified with irradiated ergosterol or 7-dehydrocholesterol such analyses do not necessarily present a true picture of the situation. Furthermore no surveys have been conducted on the liver oil of cod landed at ports in the Maritime provinces that show what factors are important in obtaining an oil of desirable potency. The results of the extensive investigation carried out by Drummond and Hilditch (4) and a later one by McPherson (6) on Newfoundland cod liver oils are related chiefly to the production of the oil and factors influencing the vitamin A potency. The production of cod liver and related topics have recently been described by Brocklesby (3). The present survey was conducted to supply information on the variations in the vitamins A and D potencies of cod liver oil and to obtain data on the factors influencing the concentration of these vitamins in the oil. In addition to providing the data it was considered

that such information would be of value to operators of processing plants and to fishermen.

The inverse relationship between the oil content of the liver and the vitamin A potency of the liver oil confirms the observations of McPherson (6) and Pugsley (8) for cod liver oil obtained from fish landed at ports in Newfoundland and in British Columbia respectively. McPherson (6) reported that the age of the fish was an important factor in determining the vitamin A potency of the liver oil and Thorbjarnarson (9) confirmed this for cod landed at ports in Iceland. The data shown in Table VIII confirm this observation and extend it to the vitamin D potency of the oil. The vitamin D potency of the liver oil of fish landed at Lockeport (Table II) is considerably higher than the values found for the other sampling places. This may possibly be due to the age factor since the fish obtained here were heavier than those from the other places. On the other hand the feeding grounds may be a factor although no significant difference was noted between the vitamins A and D potencies of the oil of inshore fish and offshore fish landed at Grand River where the feeding grounds would undoubtedly be different.

The yield of vitamins A and D per 100 gm. of fish (Table III) confirm the point that the actual concentration of the vitamins in the liver does not change with the season and the apparent seasonal variations noted are due to dilution of these constituents with oil. The higher yield of vitamin D from the fish landed at Lockeport and North Sydney as compared to Petit Rocher and Grand River is brought out here and emphasizes that the locality of catching is an important factor with respect to the yield of vitamin D.

The relation between the oil content of the liver and the stage of maturity as expressed in terms of maturation of the gonads (Tables V, VI, and VII) is very interesting and it is regretted that more data were not available on this point. Other workers have not considered this factor in surveys of the vitamins A and D potencies of the liver oil of fish. It is well known that during the spawning period fish do not feed at all or as intensively as at other times and hence during this time the stores of fat in the liver are utilized. McKenzie (5) reports that the cod off the Atlantic coast of Canada belong to a number of more or less distinct populations with regard to spawning. He divides them into two main groups (*a*) autumn spawners and (*b*) winter-spring spawners. The sampling localities chosen in this work are places that McKenzie reported as populated chiefly by winter-spring spawners. In the fish landed at Grand River the percentages of fish in Stages 1 and 2 tend to increase as the season advances from July to October and the percentages of fish in Stages 3 and 4 tend to decrease during this time. The direct relation of Stages 1 and 2 with the oil content of the liver shows that these stages are accompanied by an accumulation of fat in the liver. On the other hand the inverse relation with Stages 3 and 4 when the fish are in the immediate pre-spawning stages shows that it is during this time that the fat is being utilized by the fish.

TABLE VIII
THE EFFECT OF AGE OF FISH ON THE VITAMINS A AND D POTENCIES OF THE OIL

Date	Locality	Steaks* (six to eight years)				Market cod (four to six years)				Scrod (three to four years)			
		Oil in liver, %	Vitamin A, L value per gm. oil	Vitamin D, I.U. per gm. oil		Oil in liver, %	Vitamin A, L value per gm. oil	Vitamin D, I.U. per gm. oil		Oil in liver, %	Vitamin A, L value per gm. oil	Vitamin D, I.U. per gm. oil	
				Mean	Range $\frac{Sm}{Sm} \times 1.96$			Mean	Range $\frac{Sm}{Sm} \times 1.96$			Mean	Range $\frac{Sm}{Sm} \times 1.96$
1942													
6/6	Halifax	47.6	3000	125	81 192	48.4	1800	78	54 113	32.0	2800	44	30 64
12/6	Canso	46.8	4200	245	173 345	49.1	2100	58	41 81	49.6	1000	41	31 53
30/7	Halifax	52.7	2300	73	52 103	48.5	1000	84	67 105	38.5	4600	132	89 194
20/8	Canso	59.8	2400	118	78 178	57.2	1100	40	27 60	61.6	600	52	34 79
16/10	Mulgrave	63.7	2600	147	102 243	56.2	1100	48	33 68	55.1	1000	33	23 49

* Approximately 10% of the fish in this group are over eight years.

The increase in the oil content of the liver as the fishing season advances from June to October for the samples landed at Grand River, North Sydney, and Lockeport and the absence of any definite seasonal trend in the oil content of the liver for the samples landed at Petit Rocher may be explained on the basis of the stage of maturity of the fish. It is seen (Table VI) in the samples landed at Grand River that the majority of the fish in the samples collected during August, September, and October are in Stages 1, 2, and 3 whereas in the samples from Petit Rocher the distribution throughout the six stages is comparatively uniform during these months. Hence it appears that the oil content of the liver is governed more by stage of spawning than by season. This point deserves further study before definite conclusions can be formulated.

Acknowledgments

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REDUCTION OF SPATIAL TEMPERATURE VARIATIONS IN AIR-COOLED COLD STORAGE ROOMS. I.¹

BY T. A. STEEVES² AND W. H. COOK³

Abstract

The presence of boxes was found to increase the spatial temperature variations in a refrigerated room. By forcing the air to pass through the stack, e.g., by blocking the aisles, such variations were reduced to approximately the value prevailing in the empty room. Dunnage spacing also reduced temperature variations, but no significant difference was found between spacings varied from 5/16 in. to 2 in.

Introduction

Uniform temperatures are recognized as essential for the satisfactory storage of perishable goods in cold stores. In practice, temperature variations in time and space are common to most commercial warehouse rooms. Observations made in such rooms show that the time variations are seldom less than 1° F., and superimposed on this are the spatial variations, which are usually of greater magnitude. In fact, spatial variations of 2° F. are met frequently and still larger variations are not uncommon.

Temperature fluctuations in time are usually associated with: small storage rooms having low thermal capacity; automatic cooling equipment of an on-and-off type; faulty operation or equipment. Such variations are readily detected with a single thermometer, and since remedial measures are comparatively well understood they can generally be reduced to a reasonable level.

In contrast, spatial temperature variations are more difficult to detect and control. With only one or two thermometers placed at accessible positions in the room, many operators will deny the existence of such variations. Even if they are observed there is little that can be done to improve the situation, short of restacking and possibly redesigning the entire room. The present study was undertaken to determine the factors responsible for these spatial variations and to reduce them if possible by improved methods of air circulation.

Forced air cooling systems of modern design generally produce more uniform temperatures than the older type of convection-cooled rooms. Systems comparable with those used in air-conditioning applications usually yield satisfactory temperature uniformity in empty warehouse rooms. When the room is filled, however, the stack of products obstructs air movement and variations of detrimental magnitude frequently become evident even in the aisles. If

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the stored product is exothermic, e.g., apples, these temperature variations are enhanced further as the temperature of the air in the interior of the stack is usually higher than that of the air in the aisles.

The spatial temperature variations in a room cooled by forced air can be divided into two portions: first, a systematic increase in air temperature in the direction of flow resulting from heat absorption from the walls and product; second, irregular variations and trends resulting presumably from local peculiarities of cooling or stacking arrangements. The magnitude of the first variation is a function of the heat load and air flow, and can be reduced to any desired level in designing the room or equipment. Little is known about the factors affecting the second class of spatial variation and it is with these that the present studies are concerned.

The most generally applicable solution to the problem appears to be the development of a method that would force the air to pass through the entire width of the stack of products. This condition can best be achieved by blocking all air channels outside the stack parallel to the direction of air movement. The contents of the stack should be spaced so as to distribute the air uniformly across it, without unduly resisting the air flow. Investigations on product spacing have been made in South Africa (1, 2) in connection with the precooling of fruit. Close stacking combined with suitable baffles across open spaces resulted in higher rates of cooling, but the temperature gradients were generally higher than in "open" stacks. These results are scarcely applicable to the present problem since precooling represents a highly "unsteady" state compared with the maintenance of the desired storage temperature. British investigations (3, 4) have shown that more uniform temperature conditions are obtained during storage periods if the conventional dunnage or spacing strips are avoided.

Methods

These tests were made in an experimental room of about 700 cu. ft. capacity provided with bottom-to-top air circulation. The cool air entered through a slatted false floor and escaped through a single central port near the ceiling. Tests were made first on the empty room, then after introducing a false load of empty boxes to obstruct the air flow, and finally after introducing known heat loads by means of electrical heaters placed centrally in the first layer of boxes.

The object of adding heat was to exaggerate the spatial temperature variations and thus permit a more adequate study of the other factors under investigation rather than to study *per se* the effect of added heat. The effect of different heat loads could only be investigated under conditions that permit the control or evaluation of the portion of the temperature variations attributable to the temperature gradient in the air resulting from heat absorption. These factors had to be held over for later study. While the temperature gradient due to heat absorption by the air was included in the spatial variations as described here, the experimental conditions were chosen to minimize

the magnitude of this gradient. The room was well insulated, jacketed on the top, bottom and one side by spaces at equal or lower temperatures. A comparatively high air flow was used and the air travel through the stack was in the short vertical direction.

Effective means for blocking the vertical voids outside the stack presented a special problem. Preliminary studies showed that obstructions such as canvas dams were only partly effective since the air was merely diverted at the barrier and returned to the path of least resistance. The voids were, however, effectively closed by inflating latex-coated shelter duck bags, of suitable size, in the open spaces. In practice a series of these bags connected with hose, was inflated by means of a small auxiliary fan maintaining an air pressure of 3 to 4 in. water. The bags were readily collapsed or inflated by starting or stopping the fan, thus eliminating the need for valves.

The false load consisted of six layers of 24 boxes ($1 \times 1 \times 2$ ft.) per layer. Spacing strips were provided between each horizontal layer and also between each pair of vertical piles. Spacings of 2, $7/8$, $5/16$, and 0 in. were tested, with the surrounding vertical voids both open and closed in turn and at several levels of added heat.

The value of each experimental arrangement was assessed from observations indicating the uniformity of temperature and air flow. Measurements of air flow through the stack were made with the hot wire anemometer described previously (5). This measurement was, however, of limited applicability since the majority of the spacings chosen for study were too small to accommodate the instrument. In general, measurements could be made only above the stack and these were subject to some uncertainty since the spacings were narrower than the sensitive portion of the instrument.

The spatial temperature variations were estimated from temperature readings taken at 14 positions within the stack. The position of the thermocouples and the position of the boxes containing the heating elements are shown in Fig. 1. In addition the temperature of the air entering and leaving the room was measured, but these observations were not included in the computations. The temperature at each test position was recorded hourly on a carefully standardized recording instrument, and a single test usually extended over a 16-hr. period or longer.

The observed variations in such a series of temperature measurements include those attributable to spatial variations, fluctuations with time, and experimental errors. Every precaution was taken to maintain a constant temperature throughout the test period in order to minimize the fluctuations with time. The results of tests showing appreciable time variations were discarded. Nevertheless it was usually possible to demonstrate small but statistically significant differences between observations taken at different times for tests that were considered satisfactory. Statistical methods were therefore employed to eliminate the effect of these time variations, to obtain the standard deviation attributable to spatial variation for each test condition,

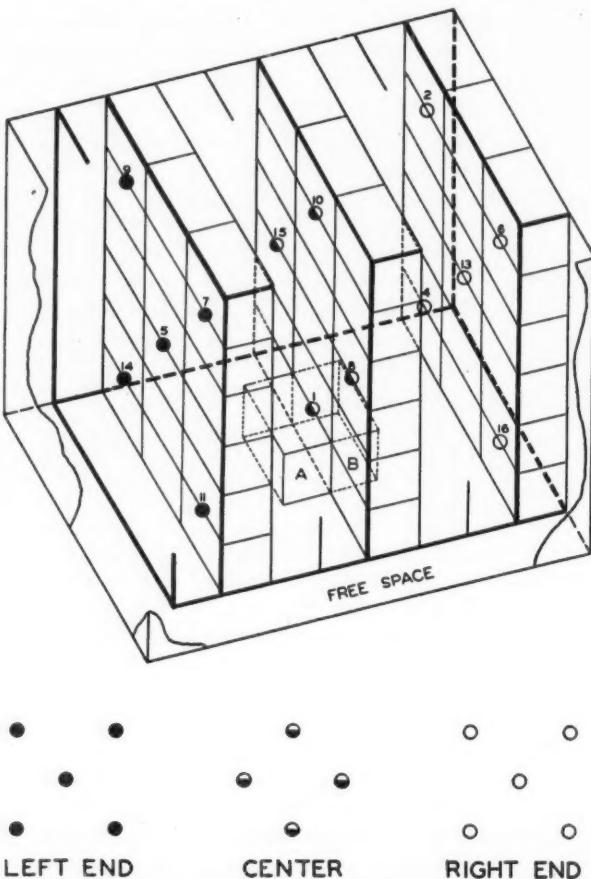


FIG. 1. Arrangement of thermocouples and boxes in test room. Numbered points indicate position of thermocouples. Boxes A and B only contained heating elements.

and to test the significance of this spatial variation by comparison with the experimental error.

When the series of experiments was complete, the standard deviations of the mean temperature, attributable to spatial variations, were themselves subjected to an analysis of variance to determine the effect of the several conditions and arrangements under test.

Results of Air Distribution Measurements

The first series of air velocity measurements was made over piles having 2-in. spacings and composed of two to five layers of boxes. In making these observations three distinct regions could be observed in the horizontal cross-

section of the room; first, the aisles or voids which were left open in these tests; second, the boxes adjacent to the aisles; and third, the interior of the stack. Measurements were made at 60 points in each of these three regions and the results grouped in this way for analysis. The limits of Regions 2 and 3 were necessarily defined arbitrarily, and were taken as being of equal area.

The average velocity for each condition tested is shown in Table I. The velocity was highest in the interior of the stack, intermediate in the region adjacent to the aisles, and least in the aisles proper. The analysis of variance shows that the observed differences were statistically significant. The addition of successive layers of boxes tended to reduce the velocity in the interior of the stack, and had a similar but smaller effect on the region adjacent to the aisles and practically no effect in the aisles themselves.

By measuring the open area available for air movement in each region described in Table I it was possible to compute the air flow for each region and for the room as a whole. Such figures are subject to some uncertainty, for in addition to the errors of estimating the air velocity and the open area involved there is also an arbitrary element introduced in definition of the borders of each region. Nevertheless the figures so obtained appear to be informative. The results showed that the regions of highest velocity actually

TABLE I
EFFECT OF PARTIAL LOADING ON AIR VELOCITY IN DIFFERENT REGIONS

Dunnage spacing, in.	Layers of boxes	Measured volume of air in circulation, c.f.m.	Horizontal region above stack		
			Vert. voids or aisles, ft./min.	Adjacent to aisles, ft./min.	Interior, ft./min.
2	2	785	21	42	63
2	3	750	20	37	55
2	4	740	19	37	47
2	5	710	18	35	41

ANALYSIS OF VARIANCE

Source of variance	Degrees of freedom	Mean square
"Areas" Residual A† "error"	2 15	6875** 608
Layers	3	256**
Areas X layers	6	64
Residual B "error"	45	28

** Exceeds 1% level of significance.

† Since the general means for the three areas were determined at a different level of precision than those for layers, the larger error "A" was used for assessing the significance of differences among areas.

received the least air, with about 45% of the total air flow passing through the aisles under all conditions. The rate of air circulation computed from the area-velocity measurements for the room as a whole accounted for about 70% of the actual air flow, as measured by other means and reported in Table I.

Since these results indicated that a large portion of the air passed through the aisles, a second series of measurements was made with the aisles both open and closed. The stack was six boxes high, and both 2 in. and 7/8 in. spacings were used. The results obtained appear in Table II. The absolute values in this table are not strictly comparable with those in Table I as some alter-

TABLE II
EFFECT OF DUNNAGE SPACING AND CLOSING AISLES ON AIR VELOCITY IN DIFFERENT REGIONS OF FULLY LOADED ROOM

Dunnage spacing, in.	Measured volume of air in circulation, c.f.m.	Horizontal region above stack		
		Vertical voids or aisles, ft./min.	Adjacent to aisles, ft./min.	Interior, ft./min.
2	760	19	34	60
2	750	Closed	62	61
7/8	700	30	34	42
7/8	670	Closed	64	62

ANALYSIS OF VARIANCE

Source of variance	Degrees of freedom	Mean square
Aisles open		
Areas (above stack)	2	2089***
Residual A† "error"	15	126
Dunnage spacing	1	50
Areas \times dunnage	2	190
Residual B "error"	15	149
Aisles closed		
Areas (above stack)	1	16
Residual A† "error"	10	50
Dunnage spacing	1	11
Areas \times dunnage	1	2
Residual B "error"	10	37

*** Exceeds 0.1% level of significance.

† Since the general means for the three areas were determined at a different level of precision than those for layers, the larger error "A" was used for assessing the significance of differences among areas.

ations were made in the equipment and stacking arrangements between these two experiments. Nevertheless with the aisles open the air velocities behaved in a manner similar to that observed in the previous experiment. When the aisles were closed the velocities in the two regions of the stack were essentially the same. The smaller spacing did not appear to increase the velocity but this may be attributable to the difficulty of measuring the velocity in a narrow space with the available anemometer.

As indicated earlier, the conversion of these velocity figures to air flow is subject to considerable error and the details are therefore not presented. These calculations showed, however, that about twice as much air passed through the aisles, when these were open, with 7/8 in. spacing as compared with 2 in. spacing in the stack. The total air flow, measured by independent means, and reported in Tables II and III shows the extent to which the closing of the aisles and the use of smaller spacing acted to reduce the air flow.

Results of Temperature Distribution Measurements

The statistical treatment of the results, described earlier, permitted the spatial variations observed for each condition or arrangement to be described as a standard deviation, independent of fluctuations in time. These values for each condition in which heat was not added appear in Table III. The

TABLE III
AVERAGE SPATIAL TEMPERATURE VARIATIONS OBSERVED UNDER VARIOUS CONDITIONS WITHOUT ADDED HEAT

Mean stack temperature, °F.	Mean air flow, c.f.m.	Vertical voids or aisles	Dunnage spacing, in.	Spatial variations as standard deviation, °F.
26	770	Empty room		0.21
34	760	Open	2	0.63
28	750	Closed	2	0.65
35	700	Open	7/8	0.50
37	670	Closed	7/8	0.48
37	675	Open	5/16	0.41
30	635	Closed	5/16	0.24
30	625	Open	0	0.97
36	500	Closed	0	0.49

complete results, including the effect of adding heat at three levels, are shown graphically in Fig. 2. Reference to Fig. 1 shows that one of the 14 thermocouples (No. 1) in the stack was adjacent to the boxes containing the heaters. Inclusion of the values obtained at this point greatly exaggerated the estimate of the spatial variations when heat was added. Since the relative results were the same this value was excluded from the computations.

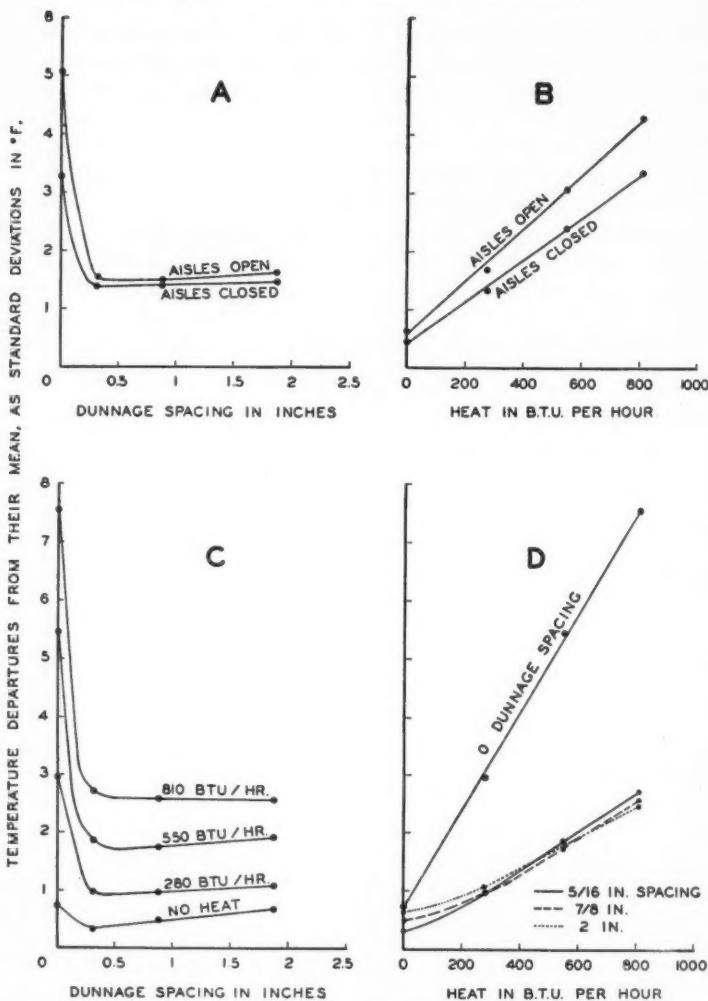


FIG. 2. *Effect of heat, dunnage and open aisles on spatial temperature variations.*

Reference to Table III shows that the spatial temperature variation in the empty room, expressed as a standard deviation, was 0.21°F . The introduction of the false load of boxes, but without added heat, caused a threefold increase to 0.63°F . This increase must be attributed entirely to the obstruction of air movement. The closest approach to the minimum deviation observed in the empty room was the value of 0.24°F . obtained at $5/16$ in. spacing with the aisles closed. The results as a whole show that the spacing must be reduced to obtain sufficient resistance to distribute the air through

the stack, and under these conditions the voids must be closed in order to force the air to traverse the pile. One complicating factor was the gradual reduction in air flow as the resistance increased. While this may have tended to increase the spatial variations by increasing the temperature gradient due to heat absorption it in no way invalidates the results. Closing the aisles was the main factor causing a reduction in air flow, but this practice actually halved the temperature variations. Had it been possible to maintain the higher air flow, the effect of closing the aisles might have been even more favourable.

The detailed results of the entire experiment are plotted in Fig. 2 and the results of an analysis of variance are given in Table IV. While this analysis shows that dunnage spacing had a significant effect on the temperature variations (Fig. 2A), a further breakdown shows that this lies entirely between dunnage versus no dunnage, and not between the individual spacings used.

TABLE IV
ANALYSIS OF VARIANCE OF SPATIAL TEMPERATURE VARIATIONS AS AFFECTED BY ALL FACTORS STUDIED

Source	Degrees of freedom	Mean square
Dunnage	3†	
Dunnage vs. no dunnage	1	43.48**
Spacings	2	0.02
Aisles (open vs. closed)	1	2.25**
Heat	3	16.64**
Dunnage \times aisles	3†	
Dunnage vs. no dunnage \times aisles	1	4.21**
Spacings \times aisles	2	.01
Dunnage \times heat	9†	
Dunnage vs. no dunnage \times heat	3	6.26**
Spacings \times heat	6	0.02
Aisles \times heat	3	0.24
Residual	9	0.14

** Exceeds 1% level of significance.

† The degrees of freedom attributable to dunnage have been partitioned as shown to illustrate that the entire significance lies between dunnage vs. no dunnage.

The analysis of variance shows that the closing of the aisles reduced the spatial temperature variations significantly at all heat levels (Fig. 2B). Added heat causes a linear increase in the standard deviations which is highly significant statistically. As previously indicated, this may be due largely to an increase in the systematic temperature gradient at the higher heat loads. The remaining significant interactions in Table IV and Fig. 2D again show that the presence or absence of dunnage is responsible for the differential behaviour noted, rather than the dunnage spacings as such.

Summary and Conclusions

The limitations of these experiments must be recognized at the outset; the room was small compared with those used in practice; the effect of air flow was not studied although it varied somewhat according to the type of test; and the system of air circulation used was bottom-to-top and not the side-to-side or end-to-end systems more commonly used in large warehouses. Further experiments are being conducted in an effort to resolve some of these factors and attempt to assess the general applicability of the results.

In spite of these limitations a few facts have been established. The introduction of a stack of product, even if it is not exothermic, increases the spatial temperature variations compared with those observed in the empty room. It follows that the observation of satisfactorily uniform temperatures in an empty room is no indication that detrimental spatial variations will not occur when the room is filled. While the temperature variations increase with increasing heat loads, as might be expected, the experiments were inadequate on this point. The effect of additional heat can only be studied under conditions that permit control or evaluation of the gradients due to heat absorption.

The air velocity measurements show substantial difference in air movement, both within the stack, and between the stack and the aisles. The indications are that in spite of a uniform initial distribution through a false floor nearly half the air moves through the aisles rather than through the stack. Closing the aisles with inflated bags rendered the air flow through the stack more uniform and reduced the spatial temperature variations. With respect to dunnage, the temperature variations increased significantly when dunnage was omitted. No significant differences in the spatial variations were observed with different dunnage spacing ranging from 5/16 to 2 in.

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AN APPARATUS AND PROCEDURE FOR STRESS CORROSION TESTING¹

By W. D. ROBERTSON²

Abstract

The design and operation of a large capacity, stress corrosion testing apparatus of the alternate immersion type for use in aluminium alloy development research is described. The testing procedure was established by investigation of the important controllable experimental variables, of which the following are discussed: (i) specimen preparation by etching in sodium hydroxide, degreasing in carbon tetrachloride, and oxide stripping with phosphoric acid inhibited by potassium dichromate; (ii) the shape of the specimen with reference to the effect of corrosion at edges as a source of erratic variation; (iii) the electrolyte concentration in both sodium chloride and hydrogen peroxide; (iv) the ratio of specimen surface to volume of electrolyte. A statistical analysis is made of the established routine procedure, and the components of variation contributed by the material and by the experimental procedure are separated and evaluated as a function of the magnitude of the corrosion. The sample size required to yield results reproducible within $\pm 10\%$ is calculated for two levels of probability, $P = 0.9$ and $P = 0.99$, for various degrees of corrosion. A table of typical results is given for different alloys in various electrolytes.

Introduction

As part of the general program of corrosion testing involving the more widely known procedures, Aluminium Laboratories Limited have developed the following apparatus to evaluate susceptibility to intercrystalline corrosion and its acceleration by stress. This type of corrosion testing finds particular application in the field of high strength alloy development research. The apparatus and procedure were designed to meet the need for a routine testing method capable of handling a large number of specimens and yielding reproducible results.

It is now generally recognized that no single accelerated test can be expected to approximate the complex system of causes from which corrosion develops in service, even if the causes are accurately known. However, the method of testing described has considerable value as a means of determining the effect of "internal" or metallurgical variables upon the susceptibility of alloys to certain well defined types of corrosion, as, for example, intercrystalline corrosion in heat treated, high strength aluminium alloys. Regarded in this light, the test may be used to advantage as a tool in alloy development work to evaluate the effect of a number of metallurgical factors such as composition, heat treatment, high temperature precipitation, and work hardening.

Therefore, in the description of apparatus and procedure that follows, the emphasis is placed on reproducibility rather than on the degree of approximation to service conditions; and no attempt is made to estimate the life of a

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material in terms of the corrosion resulting from the accelerated test beyond the fact that metallurgical conditions that lead to intergranular corrosion in the test are considered as undesirable and, as such, are to be eliminated, if possible.

Apparatus

Because stress corrosion studies constitute a large part of the work in the corrosion testing of aluminium alloys, it is necessary to provide for the application of a known stress to the material being tested and, at the same time, maintain simplicity in design and ease of operation, coupled with adequate versatility to meet the requirements of possible future work. These characteristics were specified in advance as a result of several years' experience with a similar apparatus and, after testing about 3000 specimens with the current model, shown in Fig. 1, it appears that they have been met to a very large extent.

A revolving drum, rather than a reciprocating mechanism either of an arm carrying the specimens or of the container holding the electrolyte, was adopted as the simplest method of providing for alternate immersion and emersion, since only a rotating shaft is required and there are no metallic parts in or near the electrolyte that require careful protection and servicing. The shaft is driven by a 1/6 hp. gear reduction motor in series with a second gear reducer that drives the shaft through a sprocket and chain. The motor and reducer are housed at the centre of the apparatus, Fig. 2, so that the shaft can be divided and either half driven independently at the same or at a different speed.

With the sprockets at present in use, it is possible to operate either set of five drums at any of the three speeds of two, one, or one-half revolutions per hour by transferring the driving chain from one sprocket to another located next to it. A wider range of speeds may be obtained by replacing the present set of sprockets with the required combination. One of the drums carrying the porcelain specimen holders used for stress corrosion tests is shown in Fig. 3. It is made from $\frac{1}{2}$ by 14 in. micarta discs into which are fitted the porcelain holders; the ends of the holders are fixed by an annular groove in each disc and the two discs are pushed together and locked tightly on the shaft by means of set screws in the hub. Each drum comprises 33 holders and there is provision for 10 drums, making a total capacity of 330 specimens.

The specimen holders are made of high-temperature-glazed porcelain, 1 in. wide with a $\frac{3}{8}$ in. vertical stop at each end, the inside face of which is unglazed to insure a firm contact with the end of the stressed specimen. The distance between the vertical ends of the holder is 7.5 in., and careful control in fabrication and firing has resulted in holders with an average deviation of 0.2% as determined by measurement of 100 holders.

The porcelain holders may, if desired, be replaced by the fixture shown in Fig. 3, which fits into the annular groove used for the porcelain holders and which provides for 1 in. wide unstressed strips or, alternatively, for specimens

PLATE I

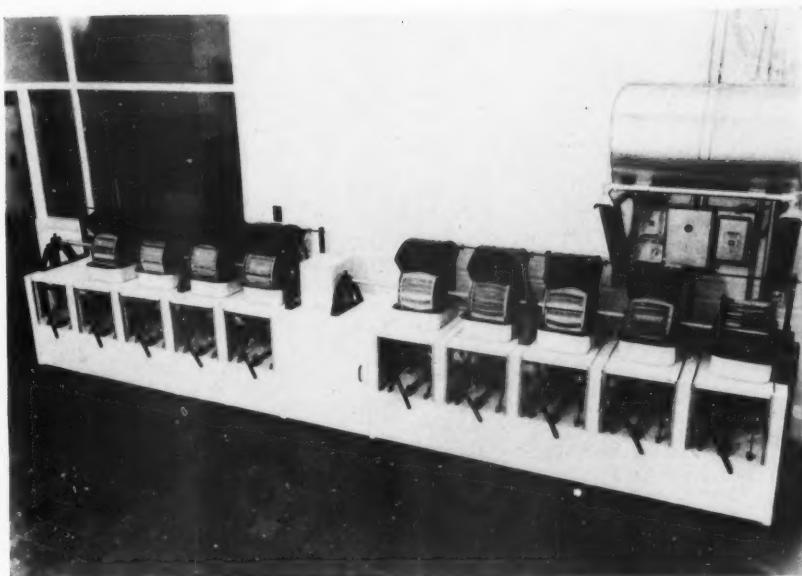


FIG. 1. *General view of rotating drum apparatus.*

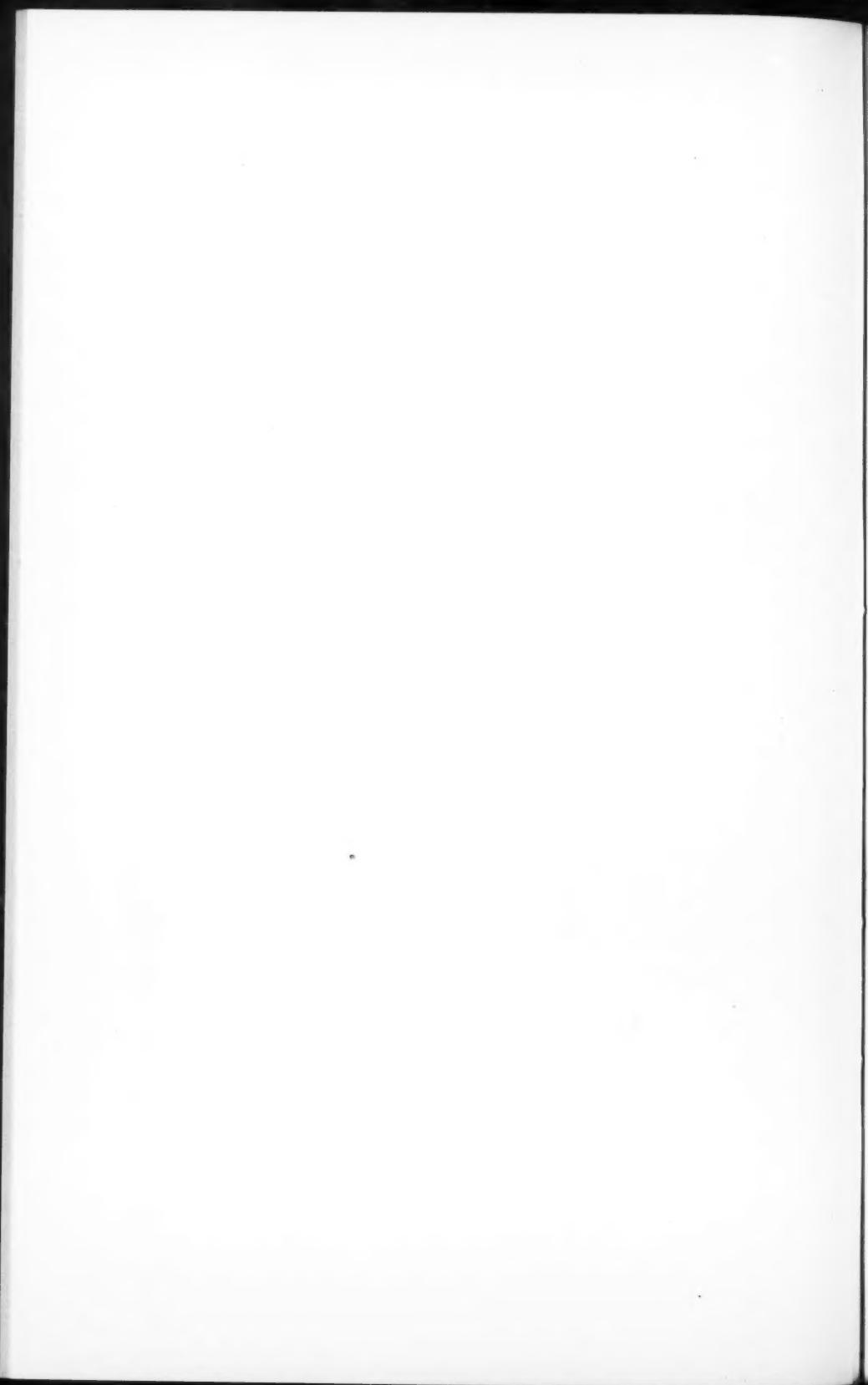


PLATE II

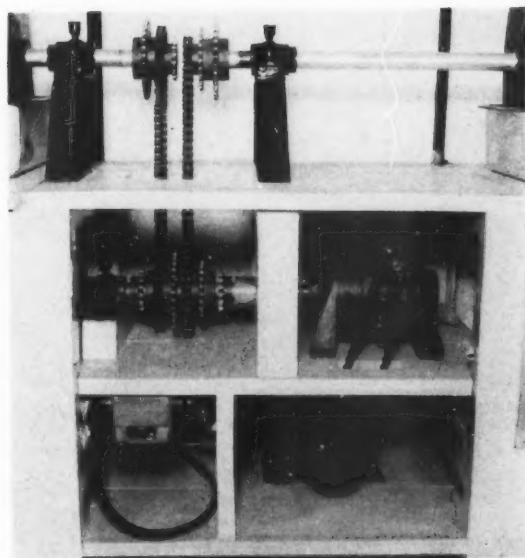


FIG. 2. *Details of driving mechanism.*

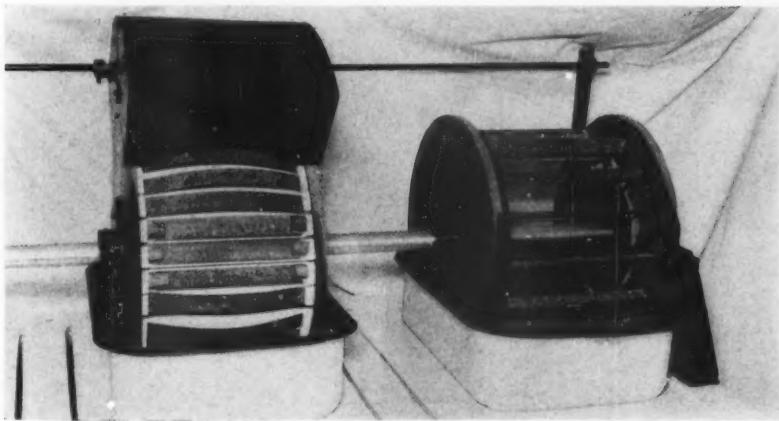
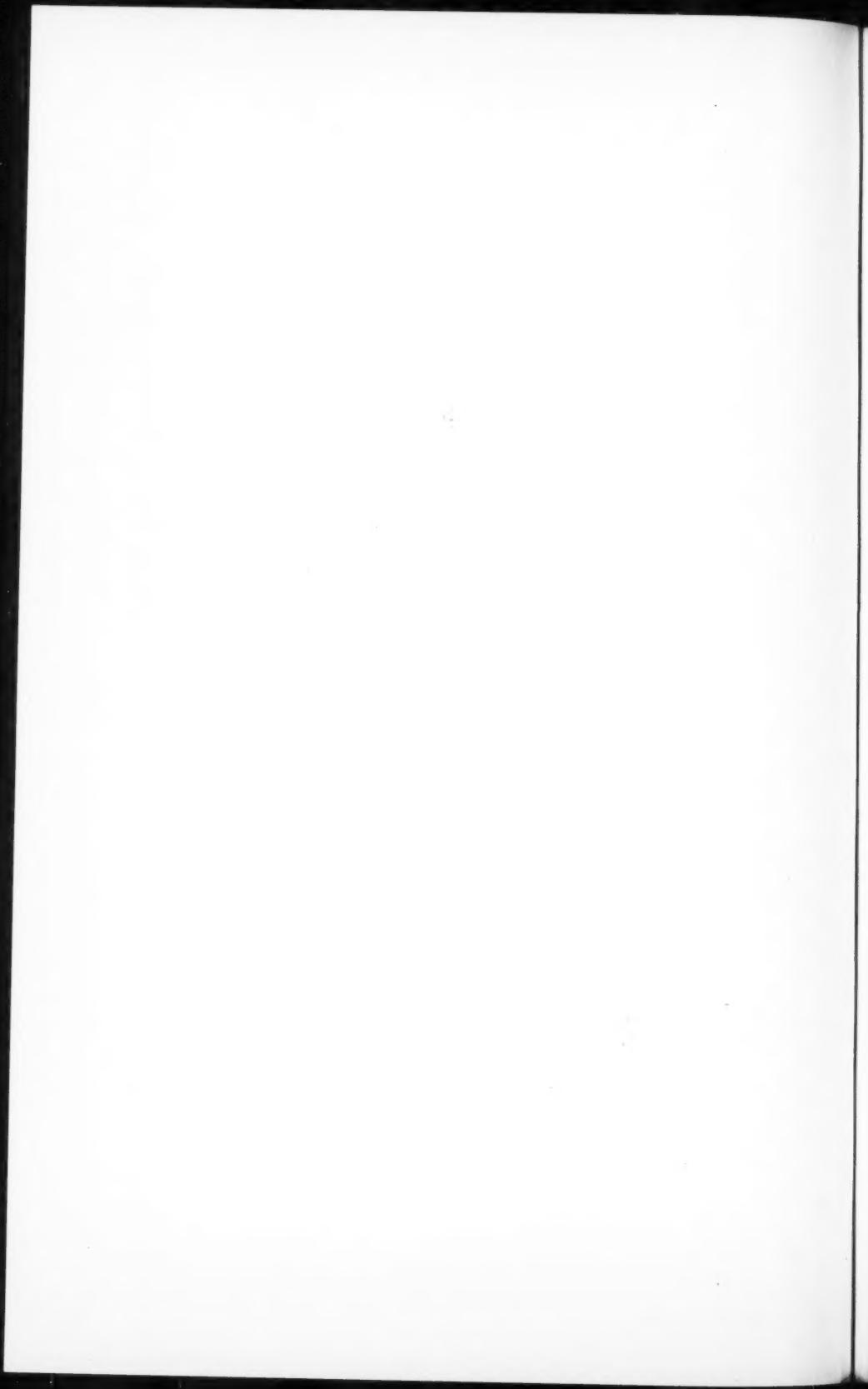


FIG. 3. *Details of drums carrying stressed and unstressed specimens.*



of various shapes suspended from horizontal rods. The fixture is divided across the diameter and is removed for loading with strip specimens by separating the two supporting discs.

Each drum revolves in a tray containing 10 litres of electrolyte, which can be lowered away from the drum for the purpose of filling or cleaning by turning the handle shown below it, thereby lowering the table under the tray. This mechanism also makes it possible to alter the distance between the tray and the drum to meet the requirements of different types of specimens.

The ratio of the time of immersion to that of atmospheric exposure depends on the geometry of the system and is 1 to 5; that is, 10 min. immersed and 50 min. exposed to the atmosphere per hour. This cycle was chosen so that adequate time would be provided for complete drying and consequent depolarization of the specimens during the cycle. However, in this connection, the room humidity is a factor to be considered, particularly with regard to seasonal changes; therefore, the lamps shown above each wheel were installed to insure drying regardless of humidity and provide independence from the prevailing atmospheric conditions and, within limits, of the speed of rotation. Each lamp holder contains four 25 watt lamps, which are sufficient to dry the specimens without raising the temperature appreciably, owing to the cooling effect of evaporation.

When conducting stress corrosion experiments, the specimens are bent in an arc between the ends of the porcelain holders. While this method does not provide uniform stress over the length of the specimen and, of course, places one side in tension and the other in compression, it has the advantage of extreme simplicity, and conditions may be easily reproduced by accurate cutting of the specimens to the required length. The apparent fibre stress can also be calculated without difficulty, though it should be emphasized that the elastic curve is not a segment of a circle but a sine curve of varying radius and, therefore, the maximum fibre stress occurs at the centre. In reality, the specimen acts more nearly as a column than a simple beam in that it is under both compressive and bending stresses, but the compressive stress is low in comparison with the bending stress. Measurements made with the tensile testing machine indicate a compressive load of about 35 lb. to maintain the curvature, which is equivalent to 700 p.s.i. on the section employed, as compared with bending stresses of the order of 40,000 to 50,000 p.s.i.

For some purposes it is desirable to run stressed and unstressed specimens simultaneously to determine the influence of stress on the corrosion, and this may be accomplished by cutting the specimens to such a length that they can be supported by inserting the ends in slotted pieces of cork and wedging the specimens and corks between the ends of the porcelain holders. The friction against the holders is sufficient to support the specimen and the compressive load required is negligible. Cork, incidentally, is preferable to rubber in hydrogen peroxide electrolytes and when wet has adequate elasticity for the purpose; in other electrolytes, rubber stoppers may be used.

After operating for one week, the drums become covered with aluminium hydroxide and it has been the practice to clean them by rotation in a solution of 1% hydrochloric acid for 24 hr., which completely dissolves the hydroxide leaving the apparatus perfectly clean.

Procedure for Corrosion Testing

The following general procedure has been adopted after a study of the various experimental factors involved. It has yielded results with consistently low deviations, and preliminary studies with standard samples indicate that large uncontrolled sources of variation have been eliminated.

Generally, the average of four to eight specimens is used to determine each point, and, when possible, the specimens are distributed over the apparatus in such a manner as to minimize the experimental variation, within the system, of variables under investigation. If the alloy is being tested for the first time and little is known regarding its stress corrosion behaviour, each point is duplicated with an equal number of specimens that are tested in the unstressed condition to define the degree of acceleration of corrosion by stress. Also, plastically deformed loops or elastically bent strips in porcelain holders may be totally immersed in a variety of electrolytes to cover the possibility of an "explosive" type of intergranular corrosion that is not disclosed by the loss in physical properties if little change takes place until immediately before the material fails.

Specimens Employed

Stress corrosion tests, in which the loss in physical properties is used as the criterion of corrosion, are usually conducted with specimens machined to the form required for subsequent physical testing (1). However, it has been found by applying the control chart technique, developed by Shewhart and outlined in the American Society for Testing Materials' Manual on the Presentation of Data (2), that the use of premachined specimens introduces a source of erratic variation such that four averages of groups of four samples each in a total of 33 groups falls outside the limits defining the permissible range of variation for a controlled experiment. Consequently, all tests are conducted with $\frac{7}{8}$ in. wide strips cut to the length required for the application of the desired bending stress, usually 80 to 90% of the yield strength, and the strips are machined to tensile specimens after the corrosion test. In this manner, work hardening and intensified corrosion at the edges and corners of specimens is eliminated, and the resulting loss in physical properties arises from the surface attack and penetration through the surface only.

For most purposes, specimens of the required length are sheared from sheet 0.060 in. thick, parallel to the rolling direction, and heat treated in baskets designed to hold them vertical during the quench. After heat treatment they are numbered at one end with the steel stamp, stored for a period sufficient to complete room temperature hardening or aged at some elevated temperature, measured for thickness and, finally, cleaned.

Specimen Preparation

Because the specimens are immersed for a comparatively short period, the method of surface preparation is of considerable importance. Consequently, three different methods of cleaning have been tried: 10% sodium hydroxide for one minute at 70° C. followed by immersion in concentrated nitric acid; degreasing with carbon tetrachloride at room temperature; and removal of the oxide film by phosphoric acid inhibited by potassium dichromate. Of the three methods, the last yields results with the lowest standard deviation (based on percentage loss in tensile strength); 0.85% for oxide stripping compared to 1.4% and 1.2% for sodium hydroxide and carbon tetrachloride respectively. Also, the degree of corrosion after oxide stripping was equal to that found after degreasing. Etching with sodium hydroxide, on the other hand, increased the degree of corrosion about 50%, of which only a small fraction could be attributed to the loss of material through the etching itself as determined by a supplementary test of etched and unetched material. To insure that no new variables were introduced by the oxide stripping, a test was made to determine the effect of the elapsed time between cleaning and the corrosion test, and it indicated that no difference exists between an immediate test and one carried out 10 days after the specimens were cleaned.

Therefore, as a result of the above test, specimens are now prepared with a solution of 20 gm. of potassium dichromate and 28 ml. of phosphoric acid (sp. gr. 1.7) per litre, for 10 min. at 70° to 80° C. In addition to the efficient cleaning and resulting low deviations, the procedure has the advantage of reducing all material, regardless of source and heat treatment, to the same basis in that it removes any film resulting from heat treatment or other production processes without destroying the surface finish characteristic of the material.

The Electrolyte

A variety of electrolytes may be used depending on the nature of the work and the alloy, but, for general testing purposes and to disclose the effect of various factors upon the susceptibility of alloys of the Al-Cu-Mg or Duralumin type to intercrystalline corrosion, a solution of 5% sodium chloride and 0.3% hydrogen peroxide in distilled water is employed. The former was chosen because it is in the range in which corrosion is independent of concentration and the latter because, at higher concentrations, the hydrogen peroxide becomes increasingly sensitive to catalytic decomposition and its rate of decomposition in contact with copper-bearing alloys is too high.

Unlike the sodium chloride, the hydrogen peroxide is decomposed during the test and the rate of decomposition is dependent on the number of specimens immersed in a given initial concentration, and on their alloy composition. It is necessary, therefore, to replenish the hydrogen peroxide, and this is done every 24 hr. during the test. The electrolyte is titrated with permanganate and the concentration made up to its initial value.

The fact that the rate of decomposition is a function of the number of specimens immersed led to an experiment to determine whether the number of specimens simultaneously tested in a constant volume of electrolyte (ratio of surface to volume of electrolyte) had an effect on the results. It was shown that, under the conditions of the above procedure, there was no appreciable difference in the degree of corrosion when the number of specimens varied from 5 to 30. It should be added, however, that this is not true in higher concentrations of hydrogen peroxide (say 3.0%), in which the corrosion decreases considerably as the number of specimens is increased beyond 10.

Criteria of Corrosion

At the conclusion of the test, usually 48 hr. in the above electrolyte or seven days in artificial sea water at one revolution per hour, the specimens are taken from the holders, the superficial corrosion product is removed with a piece of annealed aluminium, and they are cleaned again with the oxide stripping solution, and thoroughly washed. The second cleaning is necessary to protect the milling machine and cutters from corrosion by the electrolytes used in the test, and it has been ascertained that this procedure removes substantially all the chloride in so far as an analysis of distilled water in which corroded specimens were boiled showed about 10 p.p.m. of chloride, or an amount equal to that in the local water supply. It is also desirable to remove the remaining salt and inhibit further corrosion during the period that elapses between the corrosion test and the determination of the resulting physical properties.

After machining to standard test specimens, the tensile strength and elongation is measured for comparison with that found for the same material in the uncorroded condition. The degree of corrosion is then expressed as the percentage loss in tensile strength which, in reality, is a measure of the loss in effective cross-section, since the thickness is taken before the corrosion test. Unless the loss in elongation is particularly required for technical purposes, it is not used as a measure of corrosion because there is no simple relation between elongation and area as in the case of ultimate tensile strength; the latter is, of course, complicated by notching but still bears some relation to the loss of effective cross-section resulting from corrosion. It is desirable to express the results on a relative basis because the uncorroded control values frequently vary as a result of the metallurgical treatment under investigation.

If the nature of the corrosion is not already well known it may be necessary to section specimens and examine them with the microscope for intergranular corrosion. However, it cannot be too strongly emphasized that the microscope provides little in the way of information beyond a visual demonstration of intergranular attack, if it happens to be apparent in the (usually) very small area examined. The loss in physical properties can, on the other hand, be plotted and trends deduced from the resulting curve.

Analysis of Variation

The above procedure was adopted after preliminary testing of the important controllable variables. In future, standard samples will be included in each experiment to provide a continuous check on the procedure and to demonstrate that the experimental conditions are maintained in a state of control as defined by the statistical control chart (2, 3). Preliminary results, however, indicate that the procedure is under control but, for a definite statement to this effect, at least 100 standard specimens must be tested and subjected to the criterion of the statistical control chart.

To provide a measure of the deviations encountered and to determine how far the experimental procedure could be further refined to advantage, all tests performed in connection with the investigation of procedure (including about 2000 specimens) were analysed.

The analysis is based entirely on relative deviations because no single group of results included enough values to serve as a standard in the sense of the "identical" standard specimens referred to above. Consequently, deviations are expressed as a coefficient of variation, V , where

$$V = \frac{100S}{\bar{X}}.$$

S is the standard deviation in pounds per square inch, and \bar{X} , the average of the subgroup.

The coefficient of variation, V , was calculated for groups of four values, chosen at random, from the eight values obtained for each experimental point in the various investigations.

The cause of variation may be divided into two separable components; one resulting from the experimental procedure and the other from the variation inherent in the material itself and its heterogeneous character with respect to corrosion. These factors may be separated and the relative importance of each estimated by a method employed by Mears and Daniels (4) for weight loss determination.

All averages and corresponding coefficients of variation are divided into groups in accordance with the magnitude of the loss in tensile strength; that is, all those lying between zero and 1000 p.s.i. are in one group, those from 1000 p.s.i. to 2000 p.s.i. in another, and so on. The average loss in tensile strength and coefficient of variation is then calculated for each group and the values plotted as shown in Fig. 4.

It will be observed that the variation decreases rapidly as the loss in tensile strength, or degree of corrosion, increases to 10,000 p.s.i., after which it falls slowly from 10 to about 8% at 30,000 p.s.i. Thus, the curve may be divided into two parts at approximately 10% variation and 10,000 p.s.i. loss in tensile strength, and it may be shown that the part to the left of this point represents

the influence of the experimental procedure and that to the right represents the variation introduced by the material. This follows from the fact that the *absolute* value of experimental error is approximately constant, but on a relative basis it is large in comparison with the small values measured below 10,000 p.s.i.; on the other hand, the *relative* variation in material is approximately constant throughout the entire range and is consequently represented by the portion of the curve beyond 10,000 p.s.i. and has the value of the ordinate, about 8%.

A further subdivision of the source of variation may be made by separating the component introduced by the corrosion test and that arising from the material itself as it affects the results of the tensile test. Therefore, 100 "identical" uncorroded specimens were tested and the standard deviation calculated for subgroups of four to provide data on the variation encountered in the tensile test alone.

Since the criterion of corrosion, loss in tensile strength, is obtained by subtraction, the errors are retained and when the result is small the error may be relatively large. Thus, the average standard deviation in the tensile test (25 groups of 4), proved to be 610 p.s.i. and when converted to a coefficient of variation on the basis of the small values of loss in tensile strength, it appears as a large part of the over-all variation of the corrosion test as shown in Fig. 4.

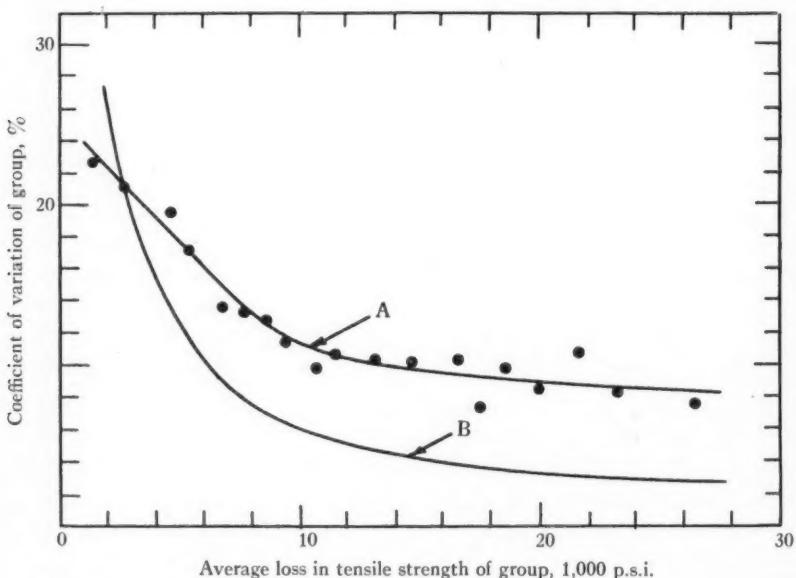


FIG. 4. *The coefficient of variation as a function of the magnitude of the values measured.* A—Experimental variation in corrosion test data. B—Variation calculated from mechanical test data.

The conclusions which may be drawn from Fig. 4 are:

- (1) Tests should be conducted for a period sufficient to result in a loss in tensile strength of at least 5000 p.s.i. so that an economical sample size may be employed to achieve the desired accuracy.
- (2) Results below 3000 p.s.i. loss in tensile strength are of doubtful value because of the effect of the material variation, which is large in comparison with the magnitude of the losses being measured in this range.
- (3) Improvements in experimental procedure probably will not result in appreciably lowering the average variations of 7 to 8% indicated for the material; they will, however, move the division point between the effect of procedure and material to lower values of the average loss in tensile strength and are warranted in this respect.
- (4) The sample size in the range of values below 10,000 p.s.i. loss in tensile strength should be larger than that used in the higher range.

From the curve of variation, the sample size required to achieve the desired accuracy may be calculated using the table of t (5) defining the probable limits of variation of averages, assuming a controlled experiment.

Therefore, if we adopt 10% as an acceptable value for limits between which the average should be found, on a probability basis, and an average standard deviation indicated by the curve, Table I shows the sample size required at various levels of corrosion resistance.

TABLE I
SAMPLE SIZE REQUIRED FOR $\pm 10\%$ LIMITS OF VARIATION IN THE AVERAGE

Loss in tensile strength, p.s.i.	Sample size ($\pm 10\%$ limit)	
	$P = 0.9$	$P = 0.99$
5000	11	25
10000	6	12
15000	5	10
20000	5	8
25000	4	8
30000	4	8

Thus, for the samples indicated, it may be said that the average loss in tensile strength will fall within $\pm 10\%$, 9 times in 10 or 99 times in 100 trials, provided the procedure is maintained in a state of control that eliminates causes of erratic variation.

Representative results obtained with different alloys in a variety of electrolytes are shown in Table II. The tests were conducted with 0.060 in. thick, longitudinal, sheet specimens, stressed to approximately 80% of their tensile yield strength. In each case, the metallurgical conditions were such that the material was least susceptible to intergranular corrosion and, consequently,

TABLE II
REPRESENTATIVE RESULTS OBTAINED WITH VARIOUS ALLOYS AND ELECTROLYTES

Alloy	Metallurgical condition		Electrolyte and time of alternate immersion at one-half rev. per hour	Corrosion	
	Solution treatment	Ageing treatment		Loss T.S., p.s.i.	Loss T.S., %
17S	505° C. cold water quench	Room temp.	48 hr. in 5% NaCl + 0.3% H ₂ O ₂	5000	8
24S	493° C. cold water quench	Room temp.	48 hr. in 5% NaCl + 0.3% H ₂ O ₂ 48 hr. in 5% NaCl + 1% HCl 48 hr. in 5% NH ₄ Cl + 0.3% H ₂ O ₂ 10 days in artificial sea water	7500 4000 10000 3000	11 6 15 4
26S	510° C. cold water quench	Room temp.	48 hr. in 5% NaCl + 0.3% H ₂ O ₂	8000	12
XA7SS	460° C. cold water quench	16 hr. at 135° C.	48 hr. in 5% NaCl + 1% HCl 10 days in artificial sea water	4000 2000	6 3

the results represent the minimum expected corrosion in the respective electrolytes; adversely altering the metallurgical conditions may increase the corrosion up to about 50% loss in tensile strength. Therefore, except for the best metallurgical conditions, and inherently high corrosion resistance, the reproducibility of the test is adequate for the purposes of a technical, routine testing method, and significant differences may be detected with comparatively small samples.

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PREPARATION OF IRISH MOSS EXTRACTS FOR USE AS A JELLING AND STABILIZING AGENT IN FOODS¹

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Abstract

Bleached Irish moss suspended on 40-mesh screens was extracted three times with water at 212° F. (100° C.). The second and third extracts were used for extracting fresh batches and by this method solutions containing 1.5 to 1.8% solids were obtained. One percent of activated charcoal mixed with the solution by agitation with air for one-half hour adsorbed all detectable flavours and odours and most of the pigment. The charcoal and suspended plant particles were removed simultaneously by filtration at 50 to 60 lb. pressure with diatomaceous earth of relatively large particle size. Potassium chloride (0.5 gm. per 100 ml.) was added to the hot filtrate, which was then poured into galvanized iron trays, allowed to gel, and frozen in air at 10° F. (-12° C.). The ice was separated mechanically from the contracted sheet of jelly, which lost on the average 90% of the water. At room temperature the rubber-like contracted sheet of jelly was dried, by means of a fan, to a residual moisture content of 8 to 10% in about two hours. The dried sheet was coarsely ground in a Wiley mill.

The resulting product employed in jellied canned chicken was preferred to an agar-agar pack by a consumer's taste panel. In grape jelly it was not a complete substitute for pectin but was considered acceptable as a fruit jelly. In three standard desserts it was not as desirable as gelatine but was considered acceptable as a jellied dessert. The material was effective in stabilizing chocolate milk in the same concentration as a commercial product now on the market.

Introduction

For many years the hot water extract (gelose) of the seaweed, Irish moss (*Chondrus crispus*), has had numerous uses. In pharmaceutical preparations it has been used as an emulsifying agent. It has been employed as a fining agent in brewing, a sizing agent in the textile industry, and a thickening agent for cold water paints.

In the food industry its chief use in recent years has been as a stabilizer for chocolate milk drinks. For this purpose 0.045% is, on the average, sufficient for satisfactory stabilization, and flavours imparted by the gelose are not detectable in the final product. The use of the extract in jellied food products was first suggested in a patent by Leon (4) who indicated that concentrations of from 1 to 2% and the presence of a potassium salt were necessary for satisfactory gels. He also stressed that for jellied food products the extract must be treated with charcoal before drying.

The claims of Leon were confirmed by Reedman and Buckby (7) who successfully used dried charcoal-adsorbed extracts as a substitute for agar-agar in canned chicken and showed that up to 0.5% the addition of potassium

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chloride to a solution of gelose increased the gel strength in a straight line relationship. However, to obtain satisfactory gels they relied solely on the natural potassium salts present in chicken broth. Their laboratory process produced a very pure extract, but, owing to the cost of drying extracts containing 0.33 to 0.46% solids, the procedure as such did not show evidence of being commercially feasible.

The object of the investigation described herein was to evolve better methods of extraction and filtration; to explore drying techniques other than spray, drum, and tunnel drying; to assess the usefulness of the product as a substitute for agar-agar, pectin, and gelatine; and to determine whether a product with high jelling properties could also be used as a stabilizer for chocolate milk drinks. The value of the material as a substitute for agar-agar was deemed most important in view of the wartime scarcity, but the production of a product of general value as a jelling and stabilizing agent was the over-all objective.

Experimental Procedure and Results

PRODUCTION OF DRIED EXTRACTS

Preparation of Sample

Dried extracts from bleached moss are lighter in colour than those from unbleached moss, and therefore more desirable for commercial purposes. Thus the data contained herein apply chiefly to sun-bleached moss from the province of Prince Edward Island. The unrefined moss as received contains sea-water salts and extraneous matter most of which can be washed off with cold water. Since Haas and Hill (3) have indicated that Irish moss contained both cold-water- and hot-water-soluble polysaccharides, experiments were made to compare the gel strengths of the two fractions.

The apparatus for determining gel strength has been described elsewhere (6). Duplicate determinations were made on quadruplicate samples of 100 ml. of gel that had been allowed to set in sealed 7 oz. lacquered cans for 18 hr. at 45° F. (7° C.).

Preliminary tests showed that a 2% solution of the alcohol precipitate of the solids extracted at 70° F. (21° C.) (the cold fraction) had approximately one-sixth the gel strength of a 2% solution of the alcohol precipitate obtained by extracting the remaining solids in water at 212° F. (100° C.) (the hot fraction). Also since the solids extracted at 70° F. (21° C.) represent a small fraction of the total extractable solids, the total fraction produces gels sufficiently strong for practical purposes. This can be seen in Fig. 1 in which the unautoclaved gel from the hot fraction has a gel strength of 118 units, whereas the gel from the total fraction has a gel strength of 105 units. After the heat treatment the samples in 7 oz. air-exhausted sealed cans were cooled immediately by the introduction of cold water into the retort. After removal from the retort the gels were handled as described above before the gel strength determinations were made.

Further, it was found that the fraction extracted at 70° F. (21° C.) had the property of stabilizing chocolate milk; and thus to obtain an extract that was satisfactory for producing gels and had maximum stabilizing ability, the total fraction, i.e. the hot and cold extract combined, was the material studied in

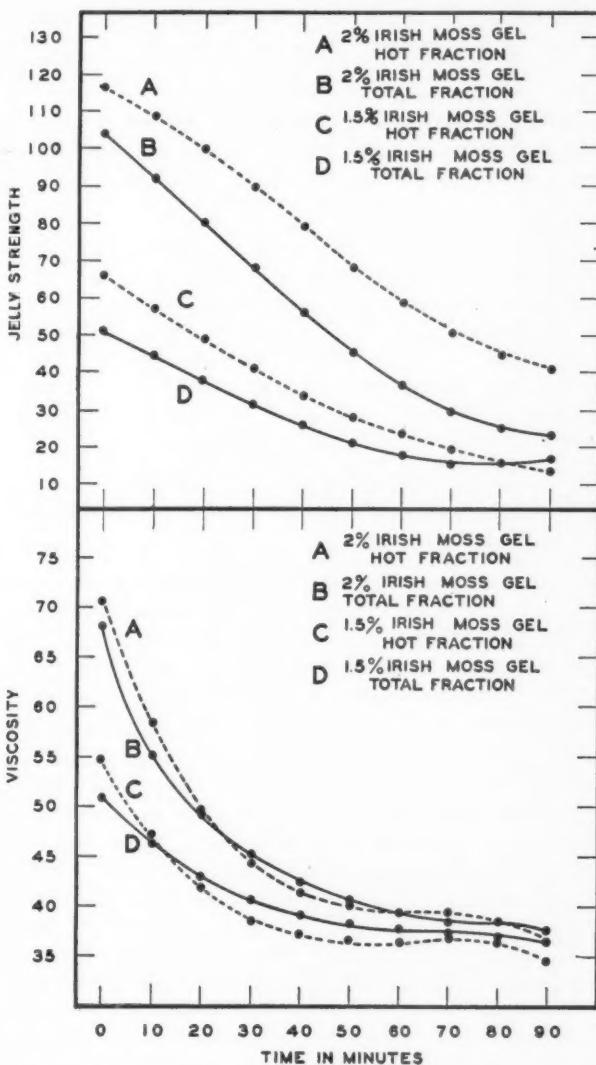


FIG. 1. The effect of a temperature of 250° F. (121° C.) on the gel strength and viscosity of two concentrations of both the total and hot fractions. The points plotted are the averages of eight measurements.

this investigation. It was found that a 10 min. preliminary spraying of the bleached moss with water at a temperature not higher than 45° F. (7° C.), washed off sand and sea water salts together with the brownish-yellow oxidized surface of bleached moss and resulted in purer, more colourless extracts; so that strictly speaking the "total fraction" alluded to above refers to the hot-water-soluble material obtainable after the moss has been thoroughly sprayed for 10 min. with water at a temperature not higher than 45° F. (7° C.).

Extraction

Extensive preliminary tests of the effects of time and temperature on the properties of gelose indicated that the conditions for extraction should be set at not longer than two and one-half hours at a temperature of 212° F. (100° C.).

Reedman and Buckby (7) obtained solutions with 0.33 to 0.47% solids. The amounts extracted were small because of filtration difficulties. These were assumed to be due to the high viscosity of the solution but in the present investigation it was found that they were caused chiefly by the concentration of insoluble suspended particles. This is illustrated by the following experiment: 6 gm. of moss was extracted in 300 ml. of hot water by vigorous agitation with a stirring motor. The larger insoluble portions of the extracted plant were removed by forcing the mixture through a 40-mesh screen, and 200 ml. of the resulting suspension containing 1% Johns-Manville No. 545 filter-aid required 95 sec. to filter through 19 sq. in. of canvas at a pressure of 20 lb. and a temperature of 200° F. (93° C.). The same volume of the filtered solution was refiltered under the same conditions in six seconds. Therefore, since the viscosity of each solution was theoretically the same, the tremendous difference in filtering time was due to the high concentration of suspended particles. It was found that if the whole plant were extracted in circulating hot water and not broken up by the agitation of a stirring motor, most of the soluble solids could be extracted and the concentration of suspended particles minimized so that filtration could be readily accomplished.

Accordingly, a cubical steam-jacketed extractor was constructed with a capacity of 53 kgm. of hot water at 212° F. (100° C.). Eight 40-mesh screens, 13.5 in. square, were soldered to $\frac{1}{2}$ in. angle-irons to form trays. These slide into the extractor on ledges at 2 in. intervals. When the gasket-covered removable end of the extractor is bolted on, the trays are forced against a gasket at the other end, to prevent liquid flowing around the ends of the trays instead of through them. The half inch contact between the angle-irons and the ledges tends to prevent the liquid flowing around the sides of the trays. The bottom of the extractor slopes from the sides and ends to an outlet at the centre. The space between the first screen and the bottom enables the liquid in the extractor to be pumped out and circulated back into the top by means of evenly spaced inlets. On each of the 40-mesh trays are placed 16-mesh screens, separated from the trays by lengths of $\frac{1}{2}$ in. wire soldered to the under surface. The coarse screens tend to keep the bulk of the moss

off the fine screens, an arrangement that prevents clogging and facilitates draining.

A quantity of dry Irish moss equal to 3% of the weight of the hot water capacity of the extractor was divided into equal portions and placed on eight trays. The moss was thoroughly sprayed with cold water as described above, evenly distributed over the surfaces of the trays, and placed in the extractor. The extractor was then filled with water at 212° F. (100° C.) and the temperature maintained by steam flowing through the jacket. The hot water was circulated through the screen-held moss for one hour. It was then pumped out. This represents the first extraction, and contains from 1.50 to 1.69% solids. However, a considerable volume of liquid fails to drain away from the moss-covered screens, so the extractor was filled from the bottom with water at 190° F. (78° C.) and extracted as before for 15 min. The combined second and third extracts, containing 0.65% solids, were heated to 212° F. (100° C.) and used to extract a fresh batch of moss weighing 2% of the hot water capacity of the extractor. This procedure readily built up a solution containing 1.8% solids after one hour at 212° F. This method gave yields of 40 to 45%.

It can thus be seen that by applying a modified counter-current system of extraction to Irish moss suspended in tanks on 40-mesh screens, readily filterable solutions containing 1.5% solids can be obtained. If screens were not used it would be impossible to circulate water through the impenetrable mass of pulp-like moss, and, as already pointed out, agitation results in filtration difficulties.

Adsorption

The extract obtained as described above has an unpleasant odour and taste, and is unsuitable for making edible gels. Taste panel tests with three dried extracts now on the market showed them unsuitable for use in jellied chicken. These products are not treated with charcoal.

In the present investigation five different charcoals were tested and Darco S-51 was selected on the basis of effectiveness, economy, and availability. It was found that this charcoal could be removed by adding 1.5% Johns-Manville No. 545 filter-aid and filtering through canvas cloths evenly precoated with a $\frac{1}{8}$ in. layer of Johns-Manville Hyflo filter-aid. This cannot be accomplished if, during adsorption, the solution is subjected to vigorous agitation, if it is passed through a centrifugal pump, or otherwise subjected to violent motion.

Accordingly, the hot solution from the extractor containing 1.6 to 1.8% solids was placed in a steam-jacketed vessel, 1 gm. of Darco S-51 per 100 ml. of solution was added and mixed for one-half hour by bubbling air into the base of the vessel. The viscous stabilizing nature of the solution tends to prevent charcoal from settling and the agitation from the air is sufficient to produce homogeneity and yet not unduly reduce the particle size of the carbon.

Filtration

The filter press used was a four-plate, steam-jacketed model of standard design with eight filtering surfaces $4\frac{1}{2}$ by $4\frac{1}{2}$ in. These were given the filter-aid precoat mentioned above; this was prevented from dropping off the cloths by blowing air at 20 lb. pressure through the press until the solution to be filtered was switched into the line at a higher pressure. If the precoat fails to cover the cloths completely the first portion of the filtered solution will be grey in colour owing to small charcoal particles, and subsequent filtering through a precoat of Hyflo filter-aid fails to remove them.

Two grams of Johns-Manville No. 545 filter-aid per 100 ml. was added to the liquid after charcoal adsorption, and thoroughly mixed by agitation with air. The mixture was allowed to flow by gravity into the blow case, and the charcoal and insoluble plant particles were filtered out simultaneously at a pressure of 50 to 60 lb. The filtered solution is tasteless and odourless but is slightly yellow in colour when viewed at depths greater than 3 in. When dried and ground the material has a slightly grey appearance indicating that probably very small amounts of charcoal remain in the solution after filtration.

Drying

The procedure finally adopted was as follows: to the hot charcoal-adsorbed, filtered liquid containing 1.6 to 1.8% solids (previously determined) was added a hot solution containing one part potassium chloride for every three parts of solids in the extract. The hot solution was stirred vigorously for five minutes to ensure complete mixing. After this addition the total solids concentration must not be lower than 2%. It is necessary to add hot potassium chloride solution to hot gelose to prevent the formation of lumps of jelly that require much heat and agitation to dissolve. With some extracts a slight precipitate formed after the addition of potassium chloride, especially if the solution remained hot for periods of longer than one-half hour. For the production of edible gels this precipitate has no practical significance, but if the material is to be used for bacteriological media the precipitate must be removed by filtration.

The extract was then poured into galvanized iron trays 33 by 20 in. to a depth of $\frac{3}{8}$ in. in a room at 45° F. (7° C.) and as free from dust as possible. The trays of clear, colourless, extremely firm jelly were then placed on wooden racks at 10° F. (-12° C.) and left in the freezing room until the temperature of the jelly was just below 32° F. (0° C.). Then a handful of crushed ice was scattered over the surface to prevent supercooling. When freezing is complete a concentrated layer of potassium chloride and gelose is sandwiched between two layers of almost pure ice. The ice can be cracked and removed leaving the rubber-like concentrated gelose-potassium-chloride sheet in one piece. Table I gives the percentage of water lost by freezing at different temperatures and depths of gel and shows that at 10° F. (-12° C.) and a depth of $\frac{3}{8}$ in. the highest percentage of water was lost. Table II shows that the resulting product contains about 80% moisture. At room temp-

TABLE I
EFFECT OF TEMPERATURE AND DEPTH OF GEL ON WATER LOST IN FREEZING

Temperature, °F.	Depth of gel, in.	Weight of jelly, oz.	Weight of wet gelose-potassium- chloride layer after ice removed, oz.	Water lost during freezing process, %
20	1/4	88	8	91
	3/8	129	12	91
	1/2	177	18	90
10	1/4	82	6	93
	3/8	130	8	94
	1/2	154	10	94
0	1/4	90	12	87
	3/8	134	34	75
	1/2	179	75	58

TABLE II
RESULTS IN DUPLICATE OF DRYING HOT SOLUTIONS CONTAINING 1.5% GELOSE AND 0.5% POTASSIUM CHLORIDE BY TWO DIFFERENT FREEZING TECHNIQUES AT 20° F. (-7° C.)

Items considered	Ice removed mechanically in the cold		Ice removed by melting in air at 70° F. (21° C.) (Japanese agar method)	
Wt. of hot solution, gm.	400	400	400	400
Wt. of jelly after setting at room temp., gm.	383	386	388	387
Wt. of tray contents after freezing, gm.	367	368	369	369
Wt. of water lost by freezing, gm.	328	328	232	218
Water lost during freezing process, %	90.8	90.3	66.5	65.0
Wt. of resulting product, gm.	37	39	134	140
Moisture content of resulting product, %	78.3	79.4	94.0	94.3
Drying time (fan, at room temp.), hr.	2 $\frac{1}{2}$	2 $\frac{1}{4}$	4 $\frac{1}{4}$	4 $\frac{1}{4}$
Wt. of dried product, gm.	8.8	8.9	7.6	7.7
Final moisture content, %	8	8	8	8
Jelly strength of 2% solution after recon- stituting, gm. of mercury	142		61	

erature the sheet was dried, by means of a fan, to 8 to 10% moisture in about two hours.

In the freezing technique described above there are two pitfalls. One is supercooling and the other the seemingly random formation of ice crystals within the concentrated sheet of jelly. If the surface of the gel is not seeded with ice crystals it becomes supercooled and when freezing occurs, micro-crystals are formed throughout the whole gel structure and mechanical separation is impossible. Continuous agitation of the trays during cooling and freezing to prevent supercooling was not as successful nor as simple as seeding with small pieces of ice.

The presence of many entrapped ice crystals causes drying difficulties: mechanical separation of the ice and jelly layers is hindered by protruding crystals, drying time is increased, and some potassium chloride and cold-water-soluble gelose is lost when the crystals melt.

The greatest single factor causing this undesirable condition is a low jelly strength that may be due either to low concentrations of gelose or potassium chloride, or to gelose that has been damaged by too much heat. Fig. 1 shows the effect of heat and time on gel strength and viscosity: gel strengths of 100 units and up were most suitable; gels weaker than 100 units had progressively more entrapped crystals; and at a gel strength less than 55 units neither the hot fraction nor the total fraction could be satisfactorily dried by the freezing technique.

Entrapped crystals were also caused by the following treatments:

Treatments	Effects
Pin-pricks in jelly surface	Crystals
Floor dust on tray before pouring sprinkled on solution before setting sprinkled on jelly after setting	Many crystals Many crystals No effect
Iron filings mixed with hot gel	Many crystals
Sawdust mixed with hot gel	Many crystals
Control	Two crystals

In the freezing technique described the potassium-chloride-gelose mixture contained 0.5% potassium chloride, because in preliminary taste panel tests it was found that above this level potassium chloride is unpleasantly salty and to some palates slightly bitter.

The goal of a 'perfect freeze', i.e. one in which no ice crystals were entrapped, occurred with two out of 20 batches even when no precautions were taken to prevent dust falling on the freshly poured trays, and depths up to $\frac{1}{2}$ in. were frozen at 0° F. (-18° C.), 10° F. (-12° C.), and 20° F. (-7° C.). These perfect batches contained 1.5% gelose and 0.5% potassium chloride and did not have an unusual jelly strength. It would seem therefore that there are unknown factors inherent in the gelose that cause variations in the number of crystals entrapped while freezing. However, the results given in Table I are representative of what is achieved on the average by the freezing technique described.

Table II compares the method of freezing and drying described above to the method used in Japan for Japanese agar. It can be seen that mechanical separation of the ice rather than the melting of the ice is essential with Irish

moss in order to prevent loss of potassium chloride and the cold-water-soluble fraction. Another advantage is that the jelly does not imbibe melted ice, which must eventually be evaporated.

After drying, the sheets were coarsely ground in a Wiley mill, for it was observed that finely ground material had a tendency to form lumps when mixed with water.

Properties of the Dried Product

This product dissolves in water at 160° F. (71° C.) without lumping and in this respect is superior to the commercial preparations now available.

A 2% gel melts between 130° F. (54° C.) and 140° F. (60° C.) and sets between 100° F. (38° C.) and 110° F. (43° C.). As shown in Fig. 1 the gel strength is slowly reduced by high temperature whereas the viscosity as measured in seconds through a 100 ml. Dudley pipette at 131° F. (55° C.) is rapidly reduced.

Perhaps the most drastic heat treatment to which the product might be subjected would be in the preparation of 6-lb. tins of jellied canned pork, which are sterilized at 230° F. (110° C.) for 200 min. Pork broth was not available; chicken broth at a lower pH was substituted, and the effect of the time-temperature conditions of the canned pork process was determined on the Irish moss preparation. Fig. 2 shows the loss in gel strength of two concentrations of commercial shredded agar and two concentrations of Irish moss extract. Under these conditions agar is more stable, yet at the end of the treatment the Irish moss gel was sufficiently strong at the 2% level to maintain its structure in air at 120° F.

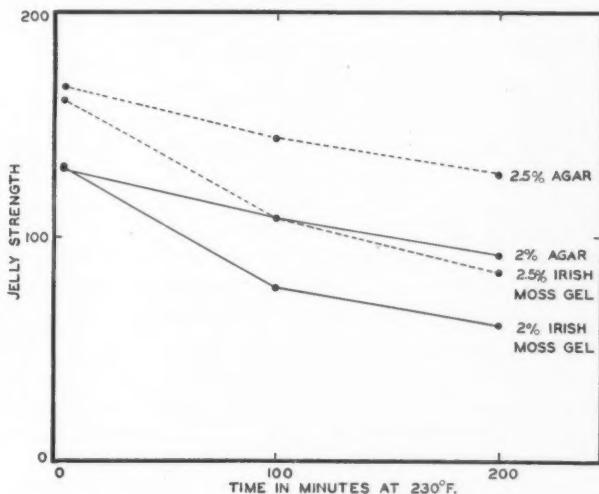


FIG. 2. The effect of prolonged heat treatment (230° F. or 110° C.) on the gel strength of two concentrations of both agar-agar and the total fraction of the Irish moss preparation.

Uses

As a Substitute for Agar-agar in Jellied Meats

Grade A milk-fed chickens were prepared by standard methods (1). After cooking, the meat was removed from the birds, pooled, and $3\frac{1}{2}$ oz. of meat, half light and half dark, packed into 7 oz. cans. One per cent of sodium chloride was added to the broth, which was then divided into two portions. To one was added 2 gm. per 100 ml. Irish moss preparation (approximately 69% gelose, 23% potassium chloride, and 8% moisture) and to the other 2 gm. per 100 ml. commercial shredded Japanese agar. Three and one-half ounce portions of each of these broths were added to cans containing meat. The cans were placed in flowing steam for 15 min., closed, and retorted 45 min. at 250° F. (121° C.).

A can of each product was given, together with a questionnaire, to 20 families, and 45 persons sampled the canned meats in their homes at meal time. The Irish moss preparation was preferred by 71%, 18% preferred agar, and 11% could find no difference. Since the gel strength of the moss preparation after retorting was weaker than that of agar, it was thought that perhaps the preference was given to Irish moss because it was softer and more reminiscent of gelatine. Accordingly, a similar pack was prepared except that the broth had $1\frac{1}{2}$ gm. of agar per 100 ml. This was compared to the Irish moss preparation by a group of 13 Dominion Government poultry inspectors: 64% preferred the moss preparation and 36% the agar preparation.

As a Substitute for Bacteriological Agar

Walker and Day (8) tested commercial 'Carragar' for its bacteriological potentialities. Since this product contained only a small amount of potassium chloride, gels as strong as agar were not obtained and the fortification of the product with a small quantity of agar was recommended.

The physical properties of the product described herein indicated two slight disadvantages for bacteriological work. One is the fact that the melting point of even a 2% solution that had been autoclaved 20 min. was between 122° F. (50° C.) and 140° F. (60° C.). This indicates that the product could not be used for the isolation of thermophiles. This disadvantage is partially balanced by the fact that unusual solid media, containing heat coagulable substances such as serum, could be remelted in a water-bath at 131° F. (55° C.) and used again for the isolation of mesophytic and psychophytic organisms, whereas the high melting point of agar makes such a procedure impossible.

Another slight disadvantage is indicated in Fig. 1. The viscosity of a $1\frac{1}{2}\%$ solution after 20 min. autoclaving is 42 sec. at 131° F. (55° C.) whereas that of $1\frac{1}{2}\%$ agar under similar conditions is 35 sec., and of water 34 sec. Thus the 'poured plate' technique would require more thorough mixing than is necessary with media containing agar, but this disadvantage is not considered serious.

A solution containing $1\frac{1}{2}\%$ Irish moss preparation, which under normal atmospheric conditions contains 8% moisture, would be composed of 1.035%

gelose and 0.345% potassium chloride. It is our opinion that this concentration of potassium chloride would not have a significant effect on the morphological or physiological characteristics of micro-organisms.

As a Substitute for Gelatine

Four gelatine desserts were prepared from standard recipes. The same desserts were prepared using the Irish moss preparation. Gelatine was preferred by 67% of 16 tasters, but all stated that the other samples were acceptable as jellied desserts. To gel 100 ml. of liquid with gelatine required 1.5 gm. To give a gel of the same volume with approximately the same strength required 0.6 gm. of the moss preparation. Tasters preferred the texture of the gelatine desserts, which seemed less brittle than those of Irish moss. On the basis of flavour and appearance there was no significant difference between the two. When the moss preparation was beaten with egg-white, and allowed to stand in the cold over-night, undesirable syneresis occurred.

As a Substitute for Pectin

Since sugar and acid are not necessary to make jelly from Irish moss, it was thought that a superior jelly with more natural fruit flavour could be prepared. Taste panel results contradicted this supposition, for the flavour produced by the combination of the high sugar and acid content of pectin jellies was preferred by 94% of 16 tasters. When 50% by weight of sucrose was added to grape juice with the moss preparation the jelly was too sweet. If the equivalent weight of citric acid found in pectin jellies was added, and the jelly boiled one minute for sterilization purposes, the moss preparation failed to gel on cooling.

In grape juice the nearest approach to the flavour of a pectin jelly was obtained by adding 30% sugar and relying on the natural acids in the juice to produce tartness. Even under these conditions 75% of 16 tasters preferred pectin jelly. However, 81% classed jellies made from Irish moss as acceptable. To gel 100 ml. of juice containing 50% by weight of sugar required 7.8 gm. of pectin crystals. To gel 100 ml. of juice containing 30% sugar by weight required 1 gm. of moss preparation or approximately one-eighth as much.

As a Stabilizer for Chocolate Milk Drinks

A comparison between the stabilizing properties of the present preparation and a dried commercial extract by a procedure described in a personal communication kindly sent by the Kraft Cheese Company, showed no difference between the two products, 0.225 gm. per 500 gm. of milk being required for complete stabilization, in both instances. Since the commercial product did not contain added potassium chloride, and had 6% moisture as compared to 8%, it would seem that the actual gelose in our preparation was superior as a stabilizer. The reason for this may be that the high temperatures used to dry the commercial extracts are detrimental, but time did not permit this point to be investigated.

Other Uses

Attempts were made to ascertain if edible solutions such as extracts of tea, coffee, or ground beef, and slurries such as finely ground beef, carrots, yeast cells, tomato juice, and orange juice could be dried by the freezing technique.

Accordingly 2 gm. of moss preparation per 100 ml. of solution was added at room temperature, thoroughly mixed, and permitted to stand for 20 min. The standing period of 20 min. caused the granules of the Irish moss preparation to swell and resulted in a shorter heating period to effect solution, a few minutes at 160° F. (71° C.) being sufficient. The solution was then poured into trays, allowed to set in the usual manner, frozen at 10° F. (-12° C.), and the concentrated sheet of solids removed and dried. All of the above-mentioned materials could be successfully dried by the freezing method except tomato juice and orange juice, which failed to separate on freezing possibly because the natural acids weakened the gel structure during the heating period.

Discussion

Of the various steps in the procedure for obtaining the Irish moss preparation perhaps the freezing technique is deserving of further elaboration. Within wide limits the behaviour of gels from Irish moss extracts on freezing corresponds to the observations on gelatine made by Moran (5) whose results correspond within narrow limits to those of Gryuner and Gorshkov (2) for the freezing of agar-agar jellies extracted from *Anfelta plicata* [sic].

Moran concluded that when gelatine gels are frozen there are two different sites of crystallization centres; those that are inside the jelly and those that are outside. The site of crystallization depended on the jelly strength and on the speed of freezing. In jellies with a gelatine content lower than 12% the internal centres of crystallization become more active. When 12% jellies were rapidly frozen by immersion in liquid air no ice formed on the surface. At temperatures from 12.2° F. (-11° C.) and up, progressively more ice formed on the surface than on the inside, until at 27.6° F. (-2° C.) a thick layer of ice formed on the surface with no ice inside.

Gels from Irish moss apparently differ from those of agar and gelatine in that, on freezing, centres of crystallization can be induced to form only on the surfaces at far lower temperatures, 0° F. (-18° C.) having been occasionally recorded as compared to 27.6° F. (-2° C.) for Moran's gelatine and 23° F. (-5° C.) for the agar used by Gryuner and Gorshkov. Since neither Moran nor the Russian workers mention taking steps to prevent supercooling, it was thought that the present method of freezing applied to agar would prevent the formation of internal centres of crystallization at lower temperatures. This, however, was not the case, although small amounts of surface ice did appear on agar gels at concentrations of 1, 1½, and 2% frozen in air at 20° F. (-7° C.). Zhelezhev (9) states that the freezing out of agar gels depends on the salt concentration, yet in our experience the addition of sodium or potassium chloride to agar gels and the application of ice crystals to prevent super-

cooling did not result in 'freezes' from which agar and ice could be readily separated without melting away the ice. Therefore it would seem that the difference between the conditions necessary for ideal freezing of Irish moss gels and those for gelatine and agar gels is due to inherent and unique properties of the polysaccharide.

If full advantage were taken of Canadian winter conditions to supplement refrigeration it is thought possible that a satisfactory jelling and chocolate milk stabilizing product could be manufactured on the commercial scale by the freezing method.

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RATION BISCUITS

III. EFFECT OF MOISTURE CONTENT ON KEEPING QUALITY¹

By J. B. MARSHALL², G. A. GRANT³, AND W. HAROLD WHITE⁴

Abstract

Ground biscuits made with two types of shortening were adjusted to moisture contents of approximately 0, 3, 6, 9, 12, 15, and 18% and stored at 43.3° C. (110° F.) for 56 wk. Keeping quality was assessed by flavour tests, peroxide oxygen, pH, and fluorescence measurements. Although the behaviour of the biscuits was similar, and variations in moisture content had small effect on flavour scores, Biscuit A, made with the more stable shortening, gave differentially higher scores at moisture levels below 6%. The evidence from the objective measurements indicated also that a moisture content of about 6% was most suitable for storage. Changes in the fat component as measured by peroxide oxygen were inhibited as the moisture content was increased over the range studied. The formation of fluorescing substances reached a maximum at a moisture content of 12% and decreased somewhat at higher levels.

Introduction

The preservation of foodstuffs by dehydration depends primarily on the reduction and control of moisture content (5, 7). In the first paper of this series it was shown that the storage life of ration biscuits containing protein-rich supplements could be extended by maintaining the moisture content below 6%. It was assumed that deterioration of the protein and carbohydrates preceded that of the fat fraction (4). However, it has been reported that more rapid deterioration of the fat occurred in milk (3) and in grain products (1) when the moisture content was reduced, and in ground cracker material stored over concentrated sulphuric acid (6). Thus for material of mixed composition, such as ration biscuits, the most satisfactory moisture content for storage should be one that achieves a balance between the rates of deterioration of the fat and non-fat components. The present study was undertaken to ascertain this optimum moisture content.

Materials and Procedure

The biscuits were prepared by a commercial manufacturer according to a simple formula consisting of 50 lb. of soft wheat flour, 5 lb. of shortening, and 6 oz. of soda. Two types of shortening were used, (A) a highly stable hydrogenated vegetable oil product and (B) a much less stable, compounded animal-vegetable shortening, the biscuits made with these shortenings being designated Biscuit A and Biscuit B respectively. Samples of the biscuits were

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ground mechanically and adjusted to moisture levels of approximately 0, 3, 6, 9, 12, 15, and 18% by the method described for dried whole egg powder (7). Calcium chloride was replaced by phosphorous pentoxide to obtain the two lowest levels. The actual values are given in Table I. Samples of about

TABLE I
MOISTURE CONTENTS OF BISCUITS

Biscuit	Moisture contents, %						
A	0.2	3.1	5.9	9.0	12.0	14.9	17.8
B	0.2	3.0	6.3	9.0	12.0	15.0	18.0

100 gm. of the adjusted material were placed in glassine bags to prevent direct contact with the tin-plate containers (No. 1 size cans) in which they were hermetically sealed, and stored at 43.3° C. (110° F.). Material for initial examination and 10 sampling times were provided for each biscuit at each of the above moisture contents.

Changes in the palatability of the material during storage were assessed by a panel of 16 tasters who scored the samples according to the following scale: 10, excellent, fresh flavour and odour; 8, good, no off-flavour or odour; 6, fair, slight off-flavour and odour; 4, poor, marked off-flavour and odour; 2, very poor, offensive flavour and odour; 0, inedible.

Objective measurements of deterioration were made by determining the peroxide oxygen values of the extracted fat and the development of fluorescing substances by methods used in previous studies of this series (3, 5). The pH of the sodium chloride extracts used in making the fluorescence measurements was also measured.

Results

The results of flavour score, fluorescence, and pH measurements are summarized in Table II to indicate the general effect of moisture content and storage time on the biscuits. The biscuit means are averages over all sample times and moisture levels; those for moisture content and storage time are the averages of the marginal totals of data summary Tables V, VI, and VII, given in the Appendix. This method of presentation provides a convenient condensation of the data for the entire experiment in a manner that shows the effects of the experimental variables. The significance of these was assessed statistically by means of analysis of variance shown in Table III.

The peroxide oxygen results given in Table IV were omitted in the summary Tables II and III as the data are not amenable to analysis of variance.

Flavour

Biscuit A made with the more stable shortening had a higher mean flavour score than Biscuit B made with the compound animal-vegetable shortening.

TABLE II
MEAN VALUES OF FLAVOUR SCORES, FLUORESCENCE, AND pH MEASUREMENTS OF BISCUITS STORED AT 43.3° C. (110° F.)

Variable	Flavour		Fluorescence		pH	
	Biscuit A	Biscuit B	Biscuit A	Biscuit B	Biscuit A	Biscuit B
Material*	6.6	6.1	39.0	36.0	7.37	7.37
Moisture, %**						
0	7.1	5.9	35.5	32.0	7.06	7.48
3	7.1	6.0	35.9	33.2	7.46	7.48
6	6.7	6.2	37.8	34.2	7.46	7.40
9	6.7	6.2	42.1	39.3	7.44	7.36
12	6.5	6.2	44.5	41.5	7.37	7.34
15	6.4	6.2	39.7	37.5	7.44	7.32
18	5.9	5.7	37.2	34.6	7.30	7.28
Time, wk.***						
Initial	8.0	7.9	34.4	30.0	7.88	8.05
2	8.0	7.2	31.1	28.2	7.51	7.61
4	7.4	6.5	41.0	36.4	7.39	7.40
6	7.2	6.6	38.3	33.5	7.24	7.21
8	7.4	6.4	40.3	36.5	7.48	7.49
12	7.2	6.3	40.0	35.6	7.54	7.54
18	6.2	5.5	34.2	30.6	7.06	7.28
24	6.4	5.9	40.0	36.9	7.44	7.46
32	6.3	6.2	44.0	43.6	6.98	6.73
44	3.3	2.9	42.4	43.4	7.02	6.92
52	5.6	5.5	43.5	41.4	7.44	7.45

* Data averaged for all moisture levels and sampling times.

** Data averaged for all sampling times.

*** Data averaged for seven moisture levels.

TABLE III
ANALYSES OF VARIANCE OF FLAVOUR SCORES, FLUORESCENCE VALUES, AND pH DATA FOR BISCUITS, ADJUSTED TO VARIOUS MOISTURE CONTENTS AND STORED AT 43.3° C. (110° F.)

Source of variance	Degrees of freedom	Mean squares		
		Flavour score	Fluorescence	pH
Biscuits	1	11.56*	333.13**	0.08
Sampling times	10	23.21**	300.74**	1.31**
Moisture levels	6		253.66**	0.11
18% vs. others	1	8.15*		
Others	5	0.18		
Biscuits × times	10	0.37*	12.05*	0.05
Biscuits × moisture levels	6	1.00**	1.28	0.17
Moisture levels × times	60	0.21**	18.65**	0.06
Biscuits × moisture levels × times	60	0.12	4.96	0.12

* Indicates 5% level of significance.

** Indicates 1% level of significance.

TABLE IV

PEROXIDE OXYGEN VALUES (ML. 0.002 N THIOSULPHATE PER GM.) OF FAT EXTRACTED FROM BISCUITS ADJUSTED TO VARIOUS MOISTURE CONTENTS AND STORED AT 43.3° C. (110° F.).

Storage time, wk.	Moisture content, %						
	0	3	6	9	12	15	18
<i>Biscuit A</i>							
Initial	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0
4	4	2	0	0	0	0	0
6	8	6	0	0	0	0	0
8	12	8	0	0	0	0	0
12	16	12	6	0	0	0	0
18	28	18	10	0	0	0	0
24	25	26	15	3	2	0	2
32	22	29	20	4	3	0	2
44	14	19	14	5	3	2	2
56	12	19	14	4	3	2	2
<i>Biscuit B</i>							
Initial	0	0	0	0	0	0	0
2	10	8	4	0	0	0	0
4	20	18	10	0	0	0	0
6	56	46	17	0	0	0	0
8	37	28	32	0	0	0	0
12	26	22	26	24	0	0	0
18	14	13	20	6	0	0	0
24	11	11	17	4	5	2	2
32	8	11	16	4	7	0	0
44	12	13	13	7	2	4	4
56	11	12	4	3	4	3	0

This difference was greater at the lower moisture levels (Fig. 1) and during the first 18 wk. of storage (Table II). Analysis of variance (Table III) showed that the two biscuits deteriorated at different rates with time and

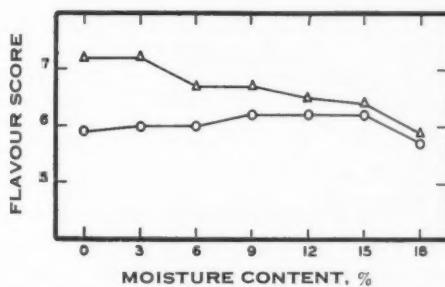


FIG. 1. The effect of moisture content on the mean flavour score. Δ = Biscuit A; \circ = Biscuit B.

moisture content. Although mean flavour scores (Table II) decreased with increasing moisture content the only significant difference was between the 18% level and the others. The anomalous flavour scores at 44 and 56 wk. may be accounted for in part by the fact that regular members of the panel were on holiday leave.

Fluorescence

The development of fluorescing substances was somewhat irregular (Table II) but followed a similar trend in both biscuits. Increasing the moisture levels resulted in maximum fluorescence values at 12% with a decline at the higher levels (Fig. 2). Grouping the data according to similarity of the trends

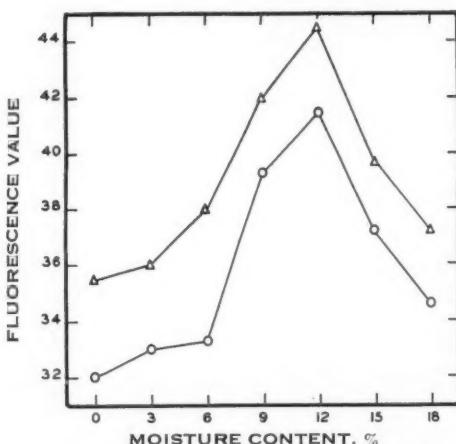


FIG. 2. *The effect of moisture content on the mean fluorescence values. △ = Biscuit A; ○ = Biscuit B.*

for the sampling times showed that initially and during the first four weeks of storage, variations in the moisture content from 0 to 12% had little effect on fluorescence values, while above this level the values progressively decreased (Fig. 3); subsequent samplings attained maximum values at 12% moisture. Analysis of variance established the significance of these trends and the differential behaviour of the Biscuits *A* and *B* during storage; the latter resulting from smaller differences between the material at the later sampling times (Table II).

With these biscuits, fluorescence measurements appear to have measured changes that were not detected by the tasters. Biscuit *A*, having the higher flavour score also had higher fluorescence values.

pH

The mean values for pH of the saline extracts shown in Table II indicate an increase of acidity with storage time, but the data were much too variable to establish significant trends.

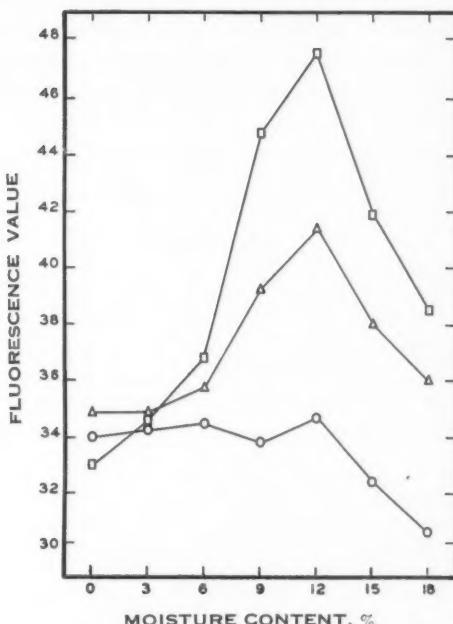


FIG. 3. The effect of moisture content on the development of fluorescence values. ○ = mean for initial, two, and four week samples; △ = mean for six and eight week samples; □ = mean for 12 to 56 week samples.

Peroxide Oxygen

Data showing the effect of moisture content on the development of peroxide oxygen in the fats are given in Table IV. The fats from Biscuit A made with the more stable vegetable oil shortening had longer induction periods than the fats made from Biscuit B at comparable moisture levels. The latter also reached much higher values than Biscuit A before decomposition of the peroxides commenced. Moisture content had a marked effect in the region of 6 to 9%; the fats from all samples of the 6% or lower being much less stable than those of the 9% or higher.

Discussion

The effect of moisture content on the deterioration in flavour of the biscuits used in this experiment was not as pronounced as the changes that were detected by objective measurements. The results have been presented without attempting to correlate objective and organoleptic data as the latter reflect the condition of the entire biscuit at the time of tasting, while peroxide oxygen values and fluorescence measurements assess changes in the fat and non-fat components. Thus while the development of peroxide oxygen values at moisture contents below 6% was accompanied by a decrease in flavour scores, flavour also deteriorated at the higher moisture levels, although peroxide

oxygen did not develop or accumulate in appreciable amounts. Changes assessed by fluorescence measurements were greater at the higher moisture levels and had reached considerable magnitude before appreciable flavour changes were detected.

The results of this investigation indicate that a moisture content of about 6% was most suitable for the storage of the biscuits.

Acknowledgments

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Appendix

TABLE V

FLAVOUR SCORES OF BISCUITS ADJUSTED TO VARIOUS MOISTURE CONTENTS AND STORED AT 43.3° C. (110° F.)

Storage time, wk.	Moisture content, %							
	0	3	6	9	12	15	18	Mean
<i>Biscuit A</i>								
Initial	7.5	8.2	8.4	8.1	7.9	8.1	7.7	8.0
2	8.3	8.4	8.1	8.1	7.7	7.9	7.2	8.0
4	8.0	8.3	7.9	7.8	1.1	7.0	6.0	7.4
6	7.8	7.9	7.4	7.3	7.3	6.5	6.1	7.2
8	7.9	7.9	7.6	7.1	7.1	7.4	6.9	7.4
12	7.9	7.1	7.1	7.2	7.0	6.3	6.6	7.2
18	6.7	6.7	5.8	6.2	5.9	5.9	5.9	6.2
24	7.4	7.4	6.4	6.2	6.0	6.4	5.1	6.4
32	6.6	6.6	6.3	6.7	6.1	6.3	5.5	6.3
44	3.9	3.9	3.4	3.4	3.5	2.6	2.4	3.3
56	5.9	5.5	5.7	5.9	5.6	5.7	5.1	5.6
Mean	7.1	7.1	6.7	6.7	6.5	6.4	6.9	6.6

TABLE V—*Concluded*FLAVOUR SCORES OF BISCUITS ADJUSTED TO VARIOUS MOISTURE CONTENTS AND STORED AT 43.3° C. (110° F.)—*Concluded*

Storage time, wk.	Moisture content, %							
	0	3	6	9	12	15	18	Mean
<i>Biscuit B</i>								
Initial	7.1	8.1	8.1	8.2	7.8	8.2	7.6	7.9
2	7.6	7.2	6.7	7.4	6.7	7.3	7.3	7.2
4	6.5	6.6	6.8	6.4	7.1	6.5	5.8	6.5
6	6.2	5.6	6.9	6.8	7.1	6.8	6.5	6.6
8	5.8	6.2	6.1	6.3	7.2	7.1	6.2	6.4
12	5.4	6.1	6.1	6.4	6.6	6.9	6.4	6.3
18	5.4	5.1	5.6	5.8	5.9	5.4	5.3	5.5
24	6.8	6.8	6.4	5.9	4.2	5.9	5.1	5.9
32	5.8	6.3	6.6	6.4	6.6	6.3	5.3	6.2
44	3.1	2.7	3.1	3.1	3.4	2.5	2.3	2.9
56	5.4	5.2	5.6	6.0	5.8	5.8	4.9	5.5
Mean	5.9	6.0	6.2	6.2	6.2	6.2	5.7	6.1

TABLE VI

FLUORESCENCE VALUES OF BISCUITS ADJUSTED TO VARIOUS MOISTURE CONTENTS AND STORED AT 43.3° C. (110° F.)

Storage time, wk.	Moisture content, %							
	0	3	6	9	12	15	18	Mean
<i>Biscuit A</i>								
Initial	34.5	34.5	34.5	34.5	35.5	34.5	32.5	34.4
2	33.5	33.0	33.5	30.5	30.5	29.5	27.0	31.1
4	39.0	41.0	41.0	40.5	45.5	40.5	38.5	41.0
6	37.5	37.0	35.5	40.0	43.5	38.5	36.0	38.3
8	36.0	38.0	39.0	41.5	46.0	41.5	40.5	40.3
12	34.5	37.5	37.5	43.5	46.5	42.0	37.5	40.0
18	27.5	24.5	35.0	42.0	40.0	37.5	32.5	34.2
24	33.0	34.0	38.0	46.0	48.0	42.0	38.0	40.0
32	40.5	41.5	41.5	45.5	52.0	42.0	44.0	44.0
44	40.0	38.0	39.0	47.0	49.0	43.0	41.0	42.4
56	34.0	36.0	41.5	52.0	53.0	46.0	42.0	43.5
Mean	35.5	35.9	37.8	42.1	44.5	39.7	37.2	39.0
<i>Biscuit B</i>								
Initial	31.5	31.5	30.0	31.5	29.5	28.5	28.0	30.0
2	31.0	30.5	30.0	26.5	27.5	26.0	26.0	28.2
4	35.0	35.0	38.0	39.5	40.0	35.5	31.5	36.4
6	32.0	32.5	32.5	35.5	35.5	35.5	31.0	33.5
8	34.0	32.0	36.0	40.0	40.5	36.5	36.5	36.5
12	32.5	32.0	33.5	34.5	42.0	38.0	36.5	35.6
18	25.0	25.0	27.0	36.0	37.0	34.0	30.0	30.6
24	31.0	32.0	35.0	41.0	43.5	39.5	36.5	36.9
32	35.0	44.5	33.0	49.5	54.5	47.0	42.0	43.6
44	32.0	38.0	34.0	52.0	56.0	49.0	43.0	43.4
56	33.0	32.0	47.0	46.0	50.0	43.0	39.0	41.4
Mean	32.0	33.2	34.2	39.3	41.5	37.5	34.6	36.0

TABLE VII

pH VALUES OF SODIUM CHLORIDE EXTRACTS OF DEFATTED BISCUITS, STORED
AT 43.3° C. (110° F.)

Storage time, wk.	Moisture content, %							
	0	3	6	9	12	15	18	Mean
<i>Biscuit A</i>								
Initial	7.59	7.78	7.78	7.81	8.04	8.31	7.86	7.88
2	6.87	7.84	8.14	7.68	7.44	7.46	7.12	7.50
4	7.34	7.66	7.65	7.73	7.15	7.18	7.02	7.39
6	7.32	7.42	7.38	7.50	7.10	7.09	6.92	7.25
8	7.18	7.58	7.42	7.58	7.50	7.52	7.62	7.48
12	7.08	7.68	7.65	7.60	7.60	7.70	7.45	7.54
18	7.18	7.15	6.92	6.65	7.02	7.20	7.32	7.06
24	7.42	7.52	7.55	7.40	7.42	7.42	7.38	7.44
32	6.05	6.98	7.02	7.22	7.25	7.22	7.12	6.98
44	6.32	7.02	7.01	7.21	7.25	7.20	7.10	7.01
56	7.32	7.49	7.52	7.48	7.32	7.57	7.41	7.44
Mean	7.06	7.46	7.46	7.44	7.37	7.44	7.30	7.37
<i>Biscuit B</i>								
Initial	8.00	8.00	7.98	8.04	8.03	8.22	8.11	8.05
2	7.83	7.77	7.98	7.62	7.42	7.38	7.28	7.61
4	7.81	7.49	7.90	7.32	7.35	7.02	6.89	7.40
6	7.38	7.42	7.42	7.19	7.10	7.12	6.82	7.21
8	7.41	7.52	7.55	7.45	7.55	7.40	7.58	7.49
12	7.48	7.70	7.49	7.51	7.60	7.62	7.42	7.54
18	7.42	7.35	6.93	7.31	7.38	7.29	7.32	7.28
24	7.40	7.43	7.50	7.50	7.52	7.45	7.40	7.46
32	6.72	7.02	6.38	6.73	6.68	6.80	6.78	6.73
44	7.28	7.05	6.52	6.83	6.73	6.92	7.12	6.92
56	7.51	7.54	7.52	7.41	7.43	7.35	7.39	7.45
Mean	7.48	7.48	7.38	7.36	7.34	7.32	7.28	7.37

